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Electronic Supplementary Information for

Triphenylphosphine-assisted dehydroxylative Csp^3-N bond formation via electrochemical oxidation

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Contents

1. General information

Reagents were purchased at the highest commercial quality grade and used without further purification, unless otherwise stated. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous material, unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC) carried out on 0.25 mm E. Merck silica plates (60F-254), using UV light (254 nm) and TLC stain with anisaldehyde-sulfuric acid for visualization. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 ºC). ^{1}H and ^{13}C NMR data were recorded with Bruker (400 MHz) or Jeol (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts were reported relative to tetramethylsilane (0 ppm for ^{1}H), $CDCl_3$ (77.0 ppm for ^{13}C). It is worth to mention that two rotamers of the products were normally observed in 2-substituted *N*-Boc-pyrrolidine derivatives.[1]

2. Experimental procedures

2.1 Electrode materials and dimensions

The instrument for undivided electrolysis is IKA® ElectraSyn 2.0 with carousel. The electrodes used in IKA® ElectraSyn 2.0 were purchased from IKA Company. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) (3.0 cm is the height of the electrode immersed in the solution) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm).

IKA ElectraSyn 2.0

IKA Carousel complete

Cyclic voltammograms were recorded on an electrochemical workstation CS150H (CorrTest®). A steady glassy carbon disk electrode (3 mm in diameter) was used as the working electrode; a platinum plate was

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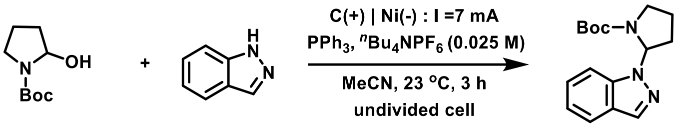
[1] a) R. E. Gawley, G. Barolli, S. Madan, M. Saverina, S. O'Connor, *Tetrahedron Lett.* **2004**, *45*, 1759–1761; b) C. P. Johnston, R. T. Smith, S. Allmendinger, D. W. C. MacMillan, *Nature* **2016**, *536*, 322–325; c) F. L. Vaillant, M. D. Wodrich, and J. Waser, *Chem. Sci.* **2017**, *8*, 1790–1800; d) D. C. Marcote, R. Street-Jeakings, E. Dauncey, J. J. Douglas, A. Ruffoni, D. Leonori, *Org. Biomol. Chem.* **2019**, *17*, 1839–1842.

used as the counter electrode; the reference was an Ag/$AgNO_3$ electrode with (0.01 M) $AgNO_3$ in acetonitrile.

2.2 General procedure for the dehydroxylative Csp^3 − N bond formation

The electrolysis was carried out in the electrolysis cell of IKA® ElectraSyn 2.0. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm). Azoles or amides (0.3 mmol), ^{n}Bu_4NPF_6 (78 mg, 0.2 mmol), alcohol (0.4 mmol), PPh_3 (105 mg, 0.4 mmol) and MeCN (8 mL) were added to an oven-dried undivided cell (10 mL) equipped with a stirring bar (the order of the addition did not affect the result). The reaction mixture was stirred and electrolyzed at a constant current of 7 mA at room temperature for 3 h. When the reaction was finished, the solvent was evaporated under vacuum and the crude material was purified by column chromatography or preparative TLC to furnish the desired product.

Table S1. Optimization of the reaction conditions[a]

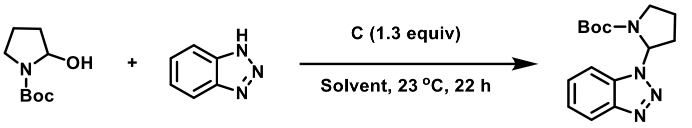


Entry[a]	Electrode	Supporting electrolyte	PR_3	Yield[b]
1	C(+) | Ni(-)	^{n}Bu_4NPF_6	PPh_3	80%
2	C(+) | C(-)	^{n}Bu_4NPF_6	PPh_3	61%
3	C(+) | Ni(-)	Et_4NPF_6	PPh_3	84%
4	C(+) | Ni(-)	^{n}Bu_4NClO_4	PPh_3	83%
5	C(+) | Ni(-)	Et_4NClO_4	PPh_3	84%
6	C(+) | Ni(-)	^{n}Bu_4NOAc	PPh_3	24%
7	C(+) | Ni(-)	^{n}Bu_4NBr	PPh_3	94%
8	C(+) | Ni(-)	^{n}Bu_4NPF_6	^{n}Bu_3P	75%
9	C(+) | Ni(-)	^{n}Bu_4NPF_6	^{t}Bu_3P	78%

10	C(+) | Ni(-)	$^{n}Bu_4NPF_6$	Tricyclohexyl Phosphine	88%
11	C(+) | Ni(-)	$^{n}Bu_4NPF_6$	Tris(4-fluorophenyl)Phosohine	95%

[a] Reaction conditions: **1a** (0.4 mmol), **2a** (0.3 mmol), PPh_3 (0.4 mmol), supporting electrolyte (0.025 M), 8 mL MeCN, 23 °C, constant current electrolysis for 3 h, open air. [b] Isolated yields after chromatography were reported.

Comparing experiments with acids.

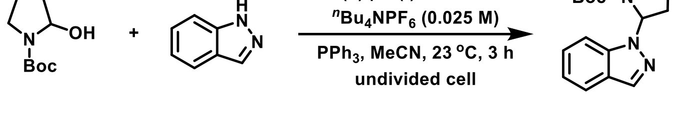


The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 1*H*-benzo[*d*][1,2,3]triazole (36 mg, 0.30 mmol) and C (0.4 mmol) at room temperature for 22 hours. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate or dichloromethane (10 mL x 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound.

C	MeCN	DCM
$BF_3 \cdot Et_2O$	-	-
$AlCl_3$	-	-
HOAc	56%	35%
CF_3CO_2H	64%	65%

3. Detail descriptions for products

Tert-butyl 2-(1*H*-indazol-1-yl)pyrrolidine-1-carboxylate (3a)

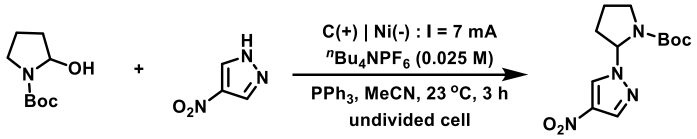


The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 1*H*-indazole (36 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 96% yield (83 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.01 (s, 1H), 7.80 – 7.45 (m, 2H), 7.41 – 7.31 (m, 1H), 7.13 (t, J = 7.5 Hz, 1H), 6.57 – 6.26 (m, 1H), 3.97 – 3.45 (m, 2H), 2.70 – 1.95 (m, 4H), 1.52 – 1.03 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 154.2, 153.5, 145.1, 139.4, 138.8, 133.5, 126.1, 125.8, 123.5, 120.7, 120.4, 109.8, 109.3, 79.9, 79.7, 69.9, 69.5, 46.5, 33.9, 32.5, 28.1, 27.9, 23.6, 22.6. **HRMS m/z (ESI)** calcd for $C_{16}H_{22}N_3O_2$ ($[M+H]^+$) 288.1707, found 288.1716.

Procedure for gram-scale synthesis: The electrolysis was carried out in the electrolysis cell of IKA® ElectraSyn 2.0. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm). According to General Procedure, starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (1.5 g, 80 mmol) was reacted with 1*H*-indazole (720 mg, 60 mmol) and PPh_3 (2.1 g, 80 mmol) were added to an oven-dried undivided cell (10 mL) equipped with a stirring bar. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA at room temperature for 24 h. Solvent was evaporated under vacuum and the crude material was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 94% yield (1.62 g).

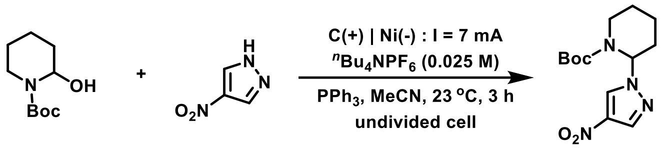
***Tert*-butyl 2-(4-nitro-1*H*-pyrazol-1-yl)pyrrolidine-1-carboxylate (3b)**



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 98% yield (83 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.37 – 8.15 (m, 1H), 8.14 – 7.99 (m, 1H), 6.06 – 5.86 (m, 1H), 3.76 – 3.36 (m, 2H), 2.48 – 1.96 (m, 4H), 1.48 – 1.31 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 154.1, 152.9, 135.8, 135.2, 128.3, 127.2, 81.4, 81.1, 74.6, 74.3, 46.9, 46.6, 33.2, 31.7, 28.1, 23.1, 22.0. **HRMS m/z (ESI)** calcd for $C_{12}H_{19}N_4O_4$ ($[M+H]^+$) 283.1401, found 283.1405.

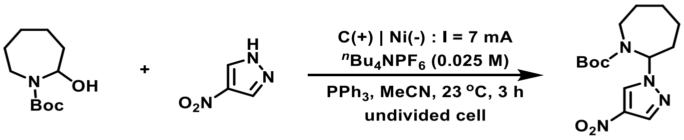
Tert-butyl 2-(4-nitro-1*H*-pyrazol-1-yl)piperidine-1-carboxylate (3c)



The starting *tert*-butyl 2-hydroxypiperidine-1-carboxylate (80 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 76% yield (68 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.17 (s, 1H), 8.09 (s, 1H), 6.31 (br s, 1H), 4.14 – 3.97 (m, 1H), 2.99 – 2.88 (m, 1H), 2.60 – 2.55 (m, 1H), 2.02 – 1.91 (m, 1H), 1.80 – 1.56 (m, 4H), 1.48 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 154.6, 135.8, 135.4, 127.6, 81.7, 67.9, 40.1, 28.2, 27.7, 24.0, 18.2. **HRMS m/z (ESI)** calcd for $C_{13}H_{21}N_4O_4$ ($[M+H]^+$) 297.1557, found 297.1562.

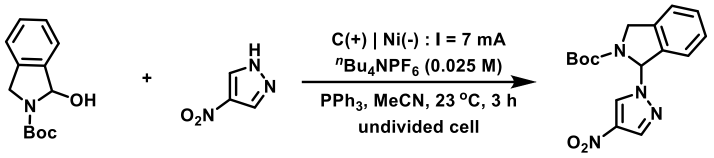
2-(4-nitro-1*H*-pyrazol-1-yl)cycloheptan-1-ol (3d)



The starting *tert*-butyl 2-hydroxyazepane-1-carboxylate (86 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 74% yield (50 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.38 – 8.13 (m, 1H), 8.12 – 8.00 (m, 1H), 6.26 – 5.98 (m, 1H), 4.06 – 3.72 (m, 1H), 3.34 – 3.20 (m, 1H), 2.51 – 2.26 (m, 2H), 2.01 – 1.36 (m, 15H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 155.5, 154.0, 135.6, 135.2, 128.4, 127.0, 81.7, 81.1, 73.5, 72.0, 42.9, 42.5, 33.4, 32.3, 29.3, 29.1, 29.1, 28.2, 28.2, 23.8, 23.6. **HRMS m/z (ESI)** calcd for $C_{13}H_{21}N_4O_4$ ($[M+H]^+$) 377.1714, found 377.1721.

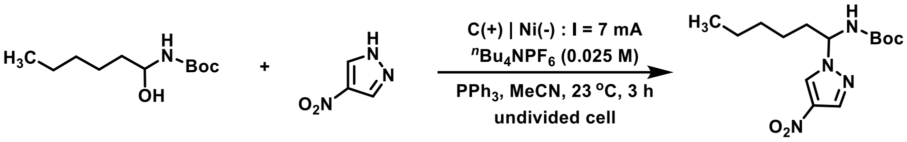
2-bromo-1-(4-nitro-1*H*-pyrazol-1-yl)isoindoline (3e)



The starting 2-bromoisoindolin-1-ol (86 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 78% yield (72 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; ^{1}**H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.44 – 8.16 (m, 1H), 8.11 – 8.00 (m, 1H), 7.51 – 7.27 (m, 4H), 7.15 – 6.98 (m, 1H), 4.94 – 4.77 (m, 2H), 1.55 – 1.38 (m, 9H). ^{13}**C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 166.6, 150.4, 140.6, 133.4, 131.4, 128.4, 124.9, 123.0, 83.1, 49.1, 28.1. **HRMS m/z (ESI)** calcd for $C_{16}H_{19}N_4O_4$ ($[M+H]^+$) 331.1401, found 331.1403.

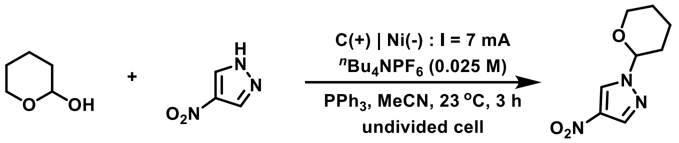
Tert-butyl (1-(4-nitro-1*H*-pyrazol-1-yl)hexyl)carbamate (3f)



The starting *tert*-butyl (1-hydroxyhexyl)carbamate (87 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 49% yield (46 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; ^{1}**H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.32 (s, 1H), 8.11 (s, 1H), 5.78 – 5.51 (m, 2H), 2.20 – 1.93 (m, 2H), 1.45 – 1.40 (m, 9H), 1.37 – 1.26 (m, 6H), 0.92 – 0.84 (m, 3H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ (ppm): 174.2, 171.1, 150.1, 82.7, 60.3, 46.4, 32.9, 27.9, 20.9, 17.3, 14.1. **HRMS m/z (ESI)** calcd for $C_{14}H_{25}N_4O_4$ ($[M+H]^+$) 313.1870, found 313.1872.

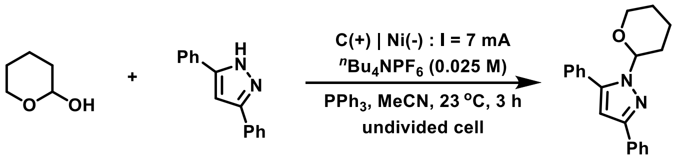
4-nitro-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole (3g)



The starting tetrahydro-2*H*-pyran-2-ol (40.8 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a colorless oil in 51% yield (30 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.34 (s, 1H), 8.07 (s, 1H), 5.44 – 5.35 (m, 1H), 4.12 – 3.66 (m, 2H), 2.22 – 1.60 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 135.4, 126.9, 88.3, 67.7, 30.6, 24.6, 21.5. **HRMS m/z (ESI)** calcd for $C_8H_{12}N_3O_3$ ($[M+H]^+$) 198.0873, found 198.0870.

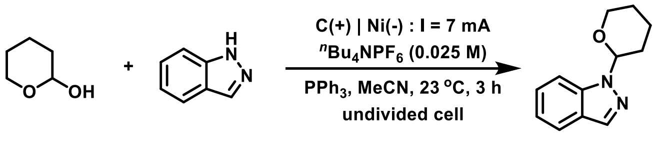
3,5-diphenyl-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole (3h)



The starting tetrahydro-2*H*-pyran-2-ol (40.8 mg, 0.4 mmol) was reacted with 3,5-diphenyl-1*H*-pyrazole (66 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a colorless oil in 61% yield (56 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 7.92 – 7.86 (m, 1H), 7.61 – 7.56 (m, 1H), 7.52 – 7.27 (m, 6H), 6.65 (s, 1H), 5.26 – 5.20 (m, 1H), 4.21 – 4.14 (m, 1H), 3.68 – 3.59 (m, 1H), 2.76 – 2.64 (m, 1H), 2.15 – 2.05 (m, 1H), 1.92 – 1.59 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 151.1, 145.5, 133.3, 130.4, 129.0, 128.6, 128.4, 127.7, 125.8, 103.7, 84.4, 67.7, 29.7, 24.8, 22.9. **HRMS m/z (ESI)** calcd for $C_{20}H_{21}N_2O$ ($[M+H]^+$) 305.1648, found 305.1652.

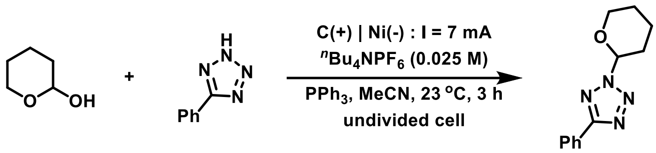
1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-indazole (3i)



The starting tetrahydro-2*H*-pyran-2-ol (40.8 mg, 0.4 mmol) was reacted with 1*H*-indazole (36 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a colorless oil in 60% yield (37 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.15 (s, 1H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.66 (d, *J* = 8.5 Hz, 1H), 7.31 – 7.25 (m, 1H), 7.11 – 7.04 (m, 1H), 5.72 – 5.64 (m, 1H), 4.18 – 3.74 (m, 2H), 2.28 – 2.16 (m, 2H), 2.11 – 1.61 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 148.5, 126.2, 121.8, 121.4, 120.9, 120.5, 117.8, 88.9, 67.9, 31.4, 24.9, 22.1. **HRMS m/z (ESI)** calcd for $C_{12}H_{15}N_2O$ ($[M+H]^+$) 203.1179, found 203.1182.

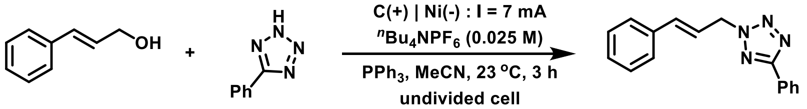
5-phenyl-2-(tetrahydro-2*H*-pyran-2-yl)-2*H*-tetrazole (3j)



The starting tetrahydro-2*H*-pyran-2-ol (40.8 mg, 0.4 mmol) was reacted with 5-phenyl-2*H*-tetrazole (44 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 70% yield (48.3 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.25 – 8.14 (m, 2H), 7.56 – 7.43 (m, 3H), 6.11 – 6.03 (m, 1H), 4.10 – 3.76 (m, 2H), 2.59 – 1.66 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 165.0, 130.3, 128.8, 127.2, 127.0, 87.8, 66.8, 29.0, 24.5, 20.8. **HRMS m/z (ESI)** calcd for $C_{12}H_{15}N_4O$ ($[M+H]^+$) 231.1240, found 231.1245.

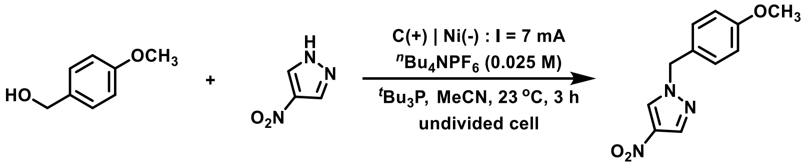
2-cinnamyl-5-phenyl-2*H*-tetrazole (3k)



The starting (*E*)-3-phenylprop-2-en-1-ol (54 mg, 0.4 mmol) was reacted with 5-phenyl-2*H*-tetrazole (44 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 31% yield (24 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.4; **^{1}H NMR** (400 MHz, CDCl$_3$) δ (ppm): 8.20 – 8.12 (m, 2H), 7.50 – 7.32 (m, 8H), 6.62 – 6.52 (m, 2H), 5.52 – 5.29 (m, 2H). **^{13}C NMR** (100 MHz, CDCl$_3$) δ (ppm): 165.1, 136.5, 133.9, 130.2, 128.9, 128.8, 127.5, 126.9, 120.1, 70.0, 29.6, 14.1. **HRMS m/z (ESI)** calcd for $C_{16}H_{15}N_4$ ([M+H]$^+$) 263.1291, found 263.1293.

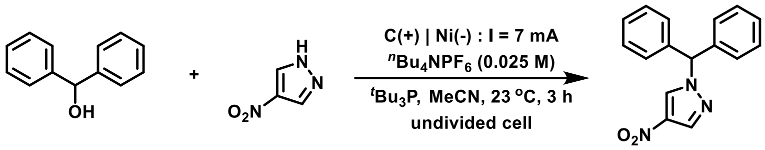
1-(4-methoxybenzyl)-4-nitro-1*H*-pyrazole (3l)



The starting (4-methoxyphenyl)methanol (55 mg, 0.4 mmol) was reacted with 4-nitro-1*H*-pyrazole (34 mg, 0.30 mmol) and ^{t}Bu$_3$P (89 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a colorless oil in 22% yield (15.3 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.3; **^{1}H NMR** (400 MHz, CDCl$_3$) δ (ppm): 8.06 (s, 1H), 7.98 (s, 1H), 7.24 (d, J = 8.3 Hz, 2H), 6.91 (d, J = 8.6 Hz, 2H), 5.22 (s, 2H), 3.81 (s, 3H). **^{13}C NMR** (100 MHz, CDCl$_3$) δ (ppm): 160.1, 135.8, 130.0, 128.0, 125.6, 114.6, 56.8, 55.3. **HRMS m/z (ESI)** calcd for $C_{11}H_{12}N_3O_3$ ([M+H]$^+$) 234.0873, found 234.0880.

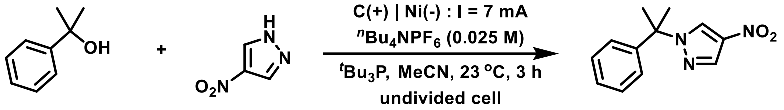
1-benzhydryl-4-nitro-1*H*-pyrazole (3m)



The starting diphenylmethanol (74 mg, 0.4 mmol) was reacted with 4-nitro-1H-pyrazole (34 mg, 0.30 mmol) and ^{t}Bu$_3$P (89 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 24% yield (20 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.2; **^{1}H NMR** (400 MHz, CDCl$_3$) δ (ppm): 8.16 (s, 1H), 7.94 (s, 1H), 7.43 – 7.34 (m, 6H), 7.14 – 7.04 (m, 4H), 6.75 (s, 1H). **^{13}C NMR** (100 MHz, CDCl$_3$) δ (ppm): 142.1, 137.5, 132.4, 130.0, 128.2, 127.2, 79.9. **HRMS m/z (ESI)** calcd for $C_{16}H_{14}N_3O_2$ ($[M+H]^+$) 280.1081, found 280.1088.

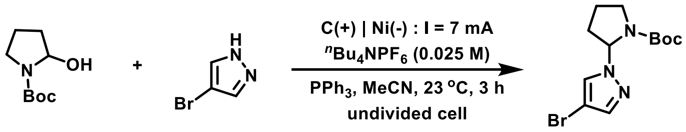
4-nitro-1-(2-phenylpropan-2-yl)-1*H*-pyrazole (3n)



The starting 2-phenylpropan-2-ol (54 mg, 0.4 mmol) was reacted with 4-nitro-1H-pyrazole (34 mg, 0.30 mmol) and ^{t}Bu$_3$P (89 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 30% yield (21 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.15 – 8.10 (m, 2H), 7.40 – 7.32 (m, 3H), 7.19 – 7.13 (m, 2H), 2.00 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 143.7, 135.3, 128.8, 128.1, 127.2, 125.1, 65.6, 29.2. **HRMS m/z (ESI)** calcd for $C_{12}H_{14}N_3O_2$ ($[M+H]^+$) 232.1081, found 232.1084.

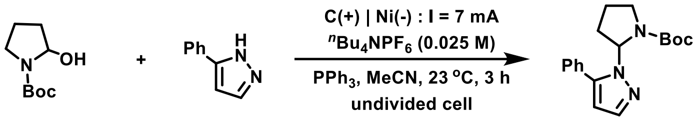
Tert-butyl 2-(4-bromo-1*H*-pyrazol-1-yl)pyrrolidine-1-carboxylate (3o)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 4-bromo-1*H*-pyrazole (44 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 83% yield (79 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.76 – 7.38 (m, 2H), 6.05 – 5.82 (m, 1H), 3.76 – 3.29 (m, 2H), 2.51 – 1.88 (m, 4H), 1.58 – 1.26 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 154.2, 153.3, 139.9, 129.3, 128.0, 92.5, 92.3, 80.8, 80.5, 73.7, 73.2, 46.8, 46.4, 33.4, 31.8, 28.2, 28.1, 23.3, 22.1. **HRMS m/z (ESI)** calcd for $C_{12}H_{19}BrN_3O_2$ ($[M+H]^+$) 316.0655, found 316.0659.

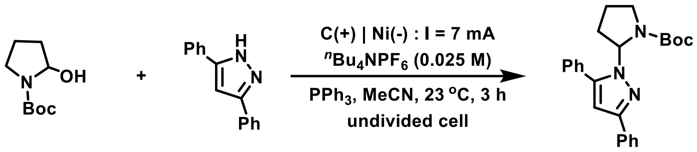
Tert-butyl 2-(5-phenyl-1*H*-pyrazol-1-yl)pyrrolidine-1-carboxylate (3p)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 5-phenyl-1H-pyrazole (44 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 70% yield (66 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.89 – 7.77 (m, 2H), 7.68 – 7.43 (m, 1H), 7.43 – 7.34 (m, 2H), 7.33 – 7.26 (m, 1H), 6.51 (s, 1H), 6.11 – 5.93 (m, 1H), 3.81 – 3.39 (m, 2H), 2.63 – 1.94 (m, 4H), 1.55 – 1.32 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 174.3, 150.2, 131.8, 128.7, 128.0, 125.7, 82.7, 46.4, 32.9, 28.0, 17.3. **HRMS m/z (ESI)** calcd for $C_{18}H_{24}N_3O_2$ ($[M+H]^+$) 314.1863, found 314.1868.

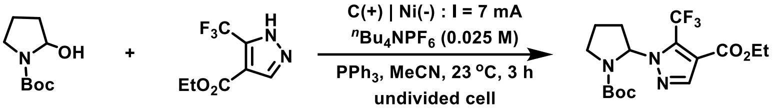
Tert-butyl 2-(3,5-diphenyl-1*H*-pyrazol-1-yl)pyrrolidine-1-carboxylate (3q)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 3,5-diphenyl-1*H*-pyrazole (66 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a white solid in 75% yield (88 mg).

Rf (petroleum ether/ethyl acetate = 10:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.91 – 7.82 (m, 2H), 7.75 – 7.25 (m, 8H), 6.65 – 6.49 (m, 1H), 6.24 – 6.03 (m, 1H), 3.99 – 3.41 (m, 2H), 2.66 – 1.84 (m, 4H), 1.49 – 1.15 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 153.9, 153.5, 150.3, 150.1, 144.6, 144.0, 133.8, 133.6, 130.8, 130.5, 129.3, 128.8, 128.6, 128.4, 128.3, 127.9, 127.4, 127.3, 125.5, 125.4, 103.2, 102.7, 80.0, 79.6, 70.4, 47.2, 47.0, 34.9, 33.6, 28.3, 28.1, 22.9, 22.0. **HRMS m/z (ESI)** calcd for $C_{24}H_{28}N_3O_2$ ($[M+H]^+$) 390.2176, found 392.2180.

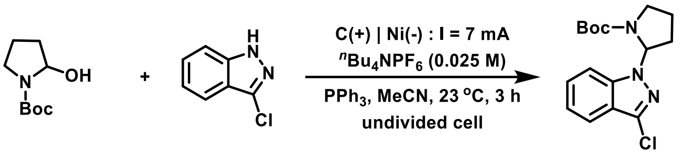
Ethyl 1-(1-(*tert*-butoxycarbonyl)pyrrolidin-2-yl)-5-(trifluoromethyl)-1*H*-pyrazole-4-carboxylate (3r)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with ethyl 5-(trifluoromethyl)-1*H*-pyrazole-4-carboxylate (63 mg, 0.30 mmol) and PPh$_3$ (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 95% yield (108 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 8.20 – 8.01 (m, 1H), 6.11 – 5.90 (m, 1H), 4.37 – 4.25 (m, 2H), 3.77 – 3.38 (m, 2H), 2.51 – 1.94 (m, 4H), 1.53 – 1.28 (m, 12H). **^{13}C NMR** (100 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 160.9 , 160.8 , 154.2 , 153.0 , 141.6 (q, *J* = 38.4 Hz), 135.1 , 133.9 , 120.3 (q, *J* = 269.2 Hz), 112.3 , 81.4 , 81.0 , 74.6 , 74.2 , 60.7 , 46.9 , 46.5 , 33.4 , 32.0 , 28.3 , 28.1 23.1 , 21.9 , 14.0. **HRMS m/z (ESI)** calcd for $C_{16}H_{23}F_3N_3O_4$ ([M+H]$^+$) 378.1635, found 378.1637.

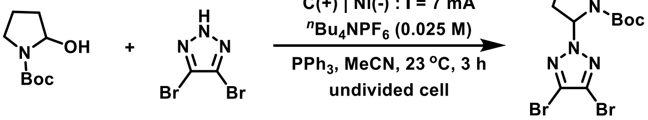
Tert-butyl 2-(3-chloro-1*H*-indazol-1-yl)pyrrolidine-1-carboxylate (3s)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 3-chloro-1*H*-indazole (46 mg, 0.30 mmol) and PPh$_3$ (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 70% yield (83 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.4; **^{1}H NMR** (400 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 7.80 – 7.37 (m, 3H), 7.24 – 7.15 (m, 1H), 6.48 – 6.19 (m, 1H), 3.95 – 3.42 (m, 2H), 2.74 – 1.92 (m, 4H), 1.48 – 1.07 (m, 9H). **^{13}C NMR** (100 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 154.3, 153.5, 140.9, 140.2, 133.5, 127.4, 127.2, 121.2, 120.7, 119.6, 119.2, 110.5, 109.8, 80.4, 80.1, 70.6, 70.0, 46.8, 46.6, 34.0, 32.6, 28.3, 28.1, 23.7, 22.6. **HRMS m/z (ESI)** calcd for $C_{16}H_{21}ClN_3O_2$ ([M+H]$^+$) 322.1317, found 322.1323.

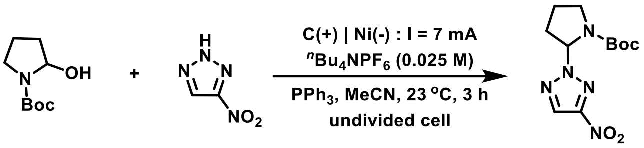
Tert-butyl *2*-(4,5-dibromo-2*H*-1,2,3-triazol-2-yl)pyrrolidine-1-carboxylate (3t)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 4,5-dibromo-2*H*-1,2,3-triazole (68 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 92% yield (109 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 6.37 – 6.07 (m, 1H), 3.87 – 3.35 (m, 2H), 2.43 – 1.89 (m, 4H), 1.53 – 1.21 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture, resonances for minor rotamer are marked with m, and resonances for major rotamer are marked with M.) δ (ppm): 153.8^m, 153.1^M, 124.4^m, 124.0^M, 81.1^M, 81.0^m, 77.7^m, 77.6^M, 46.8^m, 46.3^M, 33.4^M, 32.8^m, 28.1^m, 28.0^M, 22.5^m, 21.7^M. **HRMS m/z (ESI)** calcd for $C_{11}H_{17}Br_2N_4O_2$ ($[M+H]^+$) 396.9692, found 396.9696.

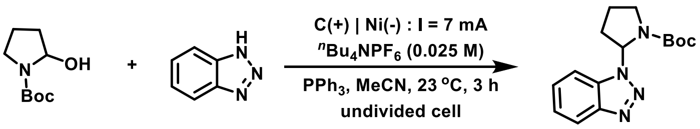
Tert-butyl 2-(4-nitro-2*H*-1,2,3-triazol-2-yl)pyrrolidine-1-carboxylate (3u)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 4-nitro-2*H*-1,2,3-triazole (34 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 83% yield (70 mg).

Rf (petroleum ether/ethyl acetate = 5:1): 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 8.17 (s, 1H), 6.50 – 6.25 (m, 1H), 3.93 – 3.45 (m, 2H), 2.56 – 1.99 (m, 4H), 1.50 – 1.25 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 153.6, 153.1, 153.0, 152.6, 130.3, 130.2, 81.1, 78.4, 46.9, 46.4, 33.6, 32.8, 28.1, 28.0, 22.5, 21.7. **HRMS m/z (ESI)** calcd for $C_{11}H_{18}N_5O_4$ ($[M+H]^+$) 284.1353, found 284.1359.

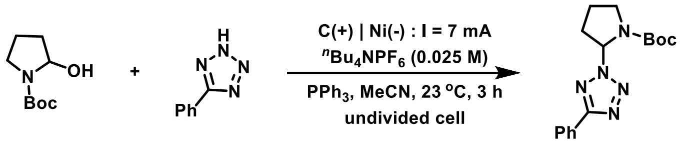
Tert-butyl 2-(1*H*-benzo[*d*][1,2,3]triazol-1-yl)pyrrolidine-1-carboxylate (3v)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 1*H*-benzo[*d*][1,2,3]triazole (36 mg, 0.30 mmol) and PPh$_3$ (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 85% yield (74 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 8.15 – 7.96 (m, 1H), 7.88 – 7.43 (m, 2H), 7.43 – 7.30 (m, 1H), 6.73 – 6.52 (m, 1H), 4.07 – 3.49 (m, 2H), 2.80 – 2.04 (m, 4H), 1.56 – 1.04 (m, 9H). **^{13}C NMR** (100 MHz, CDCl$_3$) (rotameric mixture, resonances for minor rotamer are marked with m, and resonances for major rotamer are marked with M.) δ (ppm): 154.0^{m}, 153.0^{M}, $145.5^{(m + M)}$, 132.7^{m}, 131.7^{M}, $127.1^{(m + M)}$, $123.6^{(m + M)}$, 119.8^{M}, 119.4^{m}, 110.3^{m}, 109.6^{M}, 80.7^{M}, 80.4^{m}, 71.2^{M}, 70.2^{m}, 46.7^{m}, 46.6^{M}, 34.3^{M}, 32.8^{m}, 28.1^{m}, 27.8^{M}, 23.5^{m}, 22.5^{M}. **HRMS m/z (ESI)** calcd for $C_{15}H_{21}N_4O_2$ ($[M+H]^+$) 289.1659, found 289.1667.

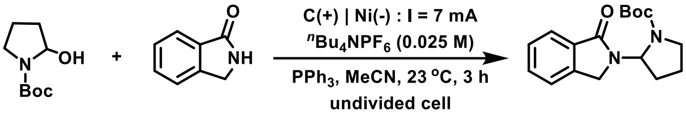
***Tert*-butyl 2-(5-phenyl-2*H*-tetrazol-2-yl)pyrrolidine-1-carboxylate (3w)**



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with 5-phenyl-2*H*-tetrazole (44 mg, 0.30 mmol) and PPh$_3$ (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 73% yield (69 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.5; **^{1}H NMR** (400 MHz, CDCl$_3$) (rotameric mixture) δ (ppm): 8.18 – 8.09 (m, 2H), 7.55 – 7.38 (m, 3H), 6.70 – 6.49 (m, 1H), 3.95 – 3.41 (m, 2H), 2.54 – 1.99 (m, 4H), 1.49 – 1.24 (m, 9H). **^{13}C NMR** (100 MHz, CDCl$_3$) (rotameric mixture, resonances for minor rotamer are marked with m, and resonances for major rotamer are marked with M.) δ (ppm): $164.7^{(m + M)}$, 153.5^{m}, 152.8^{M}, 130.1^{M}, 130.0^{m}, $128.7^{(m + M)}$, 127.5^{m}, 127.3^{M}, $126.7^{(m + M)}$, 81.1^{M}, 80.9^{m}, 75.6^{m}, 75.3^{M}, 46.7^{m}, 46.3^{M}, 33.5^{M}, 32.8^{m}, 28.1^{m}, 28.0^{M}, 22.6^{m}, 21.9^{M}. **HRMS m/z (ESI)** calcd for $C_{16}H_{22}N_5O_2$ ($[M+H]^+$) 316.1768, found 316.1772.

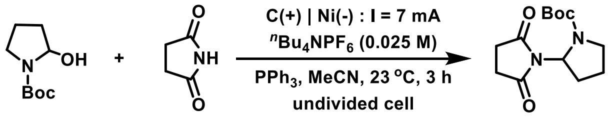
***Tert*-butyl 2-(1-oxoisoindolin-2-yl)pyrrolidine-1-carboxylate (3x)**



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with isoindolin-1-one (40 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 58% yield (53 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 7.89 – 7.81 (m, 1H), 7.61 – 7.51 (m, 1H), 7.50 – 7.41 (m, 2H), 6.13 (s, 1H), 4.31 (s, 2H), 3.67 – 3.45 (m, 2H), 2.37 – 1.93 (m, 4H), 1.56 – 1.15 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 167.9, 154.1, 140.1, 132.4, 131.4, 128.0, 123.6, 122.7, 80.2, 65.0, 46.5, 45.5, 32.4, 28.1, 23.0. **HRMS m/z (ESI)** calcd for $C_{17}H_{23}N_2O_3$ ($[M+H]^+$) 303.1703, found 303.1708.

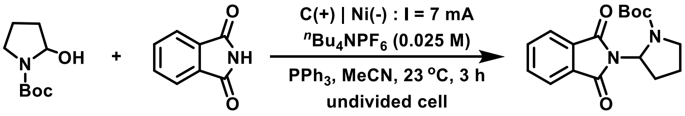
Tert-butyl 2,5-dioxo-[1,2'-bipyrrolidine]-1'-carboxylate (3y)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with pyrrolidine-2,5-dione (30 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 65% yield (52 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 5.87 – 5.72 (m, 1H), 3.64 – 3.38 (m, 2H), 2.68 – 2.55 (m, 4H), 2.32 – 1.76 (m, 4H), 1.41 – 1.31 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 176.5, 175.9, 153.4, 152.9, 80.0, 79.8, 64.6, 64.2, 47.2, 46.9, 31.2, 30.5, 28.4, 28.2, 27.9, 23.9, 23.5. **HRMS m/z (ESI)** calcd for $C_{13}H_{21}N_2O_4$ ($[M+H]^+$) 269.1496, found 269.1500.

Tert-butyl 2-(1,3-dioxoisoindolin-2-yl)pyrrolidine-1-carboxylate (3z)

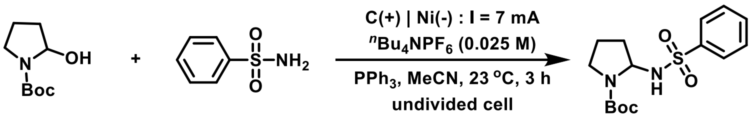


The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with isoindoline-1,3-dione (44 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure.

Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a white solid in 65% yield (62 mg).

Rf (petroleum ether/ethyl acetate = 1:1): 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.91 – 7.78 (m, 2H), 7.78 – 7.66 (m, 2H), 6.12 – 5.90 (m, 1H), 3.79 – 3.44 (m, 2H), 2.46 – 1.85 (m, 4H), 1.40 – 1.21 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 167.6, 167.3, 153.6, 153.2, 134.1, 133.8, 131.9, 131.8, 123.2, 123.1, 80.1, 79.8, 63.9, 63.6, 47.2, 46.8, 32.0, 31.3, 28.2, 23.9, 23.4. **HRMS m/z (ESI)** calcd for $C_{17}H_{21}N_2O_4$ ($[M+H]^+$) 317.1496, found 317.1505.

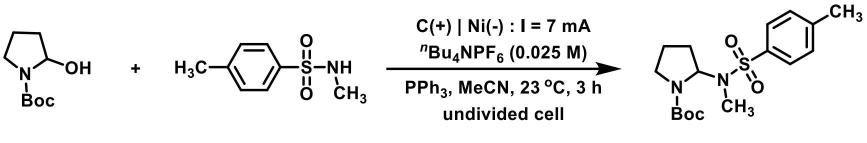
Tert-butyl 2-(phenylsulfonamido)pyrrolidine-1-carboxylate (3aa)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with benzenesulfonamide (47 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to afford the title compound as a white solid in 36% yield (35 mg).

Rf (petroleum ether/ethyl acetate = 1:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.99 – 7.83 (m, 2H), 7.64 – 7.45 (m, 3H), 5.62 – 4.89 (m, 2H), 3.43 – 3.12 (m, 2H), 2.39 – 1.78 (m, 4H), 1.42 – 1.25 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 153.9, 153.3, 141.0, 140.5, 132.4, 128.9, 126.9, 126.6, 80.6, 80.1, 68.0, 45.9, 45.5, 32.9, 31.5, 28.1, 22.5, 21.4. **HRMS m/z (ESI)** calcd for $C_{15}H_{23}N_2O_4S$ ($[M+H]^+$) 327.1373, found 327.1380.

Tert-butyl 2-((*N*,4-dimethylphenyl)sulfonamido)pyrrolidine-1-carboxylate (3ab)

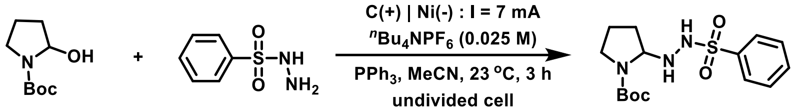


The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with *N*,4-dimethylbenzenesulfonamide (56 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3).

The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to afford the title compound as a white solid in 85% yield (90 mg).

Rf (petroleum ether/ethyl acetate = 1:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) (rotameric mixture) δ (ppm): 7.83 – 7.60 (m, 2H), 7.38 – 7.23 (m, 2H), 5.86 – 5.17 (m, 1H), 3.56 – 3.18 (m, 2H), 2.67 – 2.54 (m, 3H), 2.47 – 2.37 (m, 3H), 2.14 – 1.75 (m, 4H), 1.53 – 1.41 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) (rotameric mixture) δ (ppm):154.0, 153.9, 143.1, 142.8, 136.5, 135.7, 129.4, 127.0, 81.4, 79.8, 70.9, 45.7, 33.3, 32.6, 29.0, 28.3, 28.2, 28.1, 27.8, 22.5, 21.3. **HRMS m/z (ESI)** calcd for $C_{17}H_{27}N_2O_4S$ ($[M+H]^+$) 355.1686, found 355.1693.

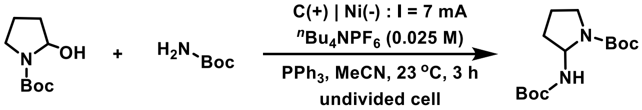
Tert-butyl 2-(2-(phenylsulfonyl)hydrazinyl)pyrrolidine-1-carboxylate (3ac)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with benzenesulfonohydrazide (52 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a white solid in 35% yield (36 mg).

Rf (petroleum ether/ethyl acetate = 1:1): 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 7.98 – 7.89 (m, 2H), 7.63 – 7.49 (m, 3H), 4.50 – 4.36 (m, 1H), 3.37 – 3.11 (m, 2H), 1.93 – 1.62 (m, 4H), 1.50 – 1.42 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 156.0, 138.2, 132.8, 128.7, 128.1, 80.3, 73.5, 46.7, 29.8, 28.3, 23.1. **HRMS m/z (ESI)** calcd for $C_{15}H_{24}N_3O_4S$ ($[M+H]^+$) 342.1482, found 342.1490.

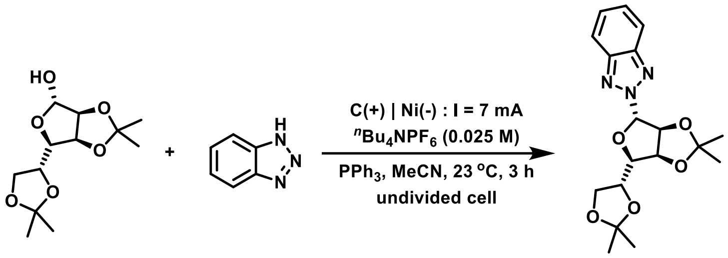
Tert-butyl 2-((tert-butoxycarbonyl)amino)pyrrolidine-1-carboxylate (3ad)



The starting *tert*-butyl 2-hydroxypyrrolidine-1-carboxylate (75 mg, 0.4 mmol) was reacted with *tert*-butyl carbamate (35 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a colorless oil in 49% yield (42 mg).

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 5.40 (br s, 1H), 4.78 (br s, 1H), 3.55 – 3.14 (m, 2H), 2.14 – 1.76 (m, 4H), 1.46 (s, 9H), 1.44 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 154.2, 154.1, 79.9, 79.3, 65.6, 45.8, 34.2, 28.4, 28.4, 22.5. **HRMS m/z (ESI)** calcd for $C_{14}H_{27}N_2O_4$ ($[M+H]^+$) 287.1965, found 287.1972.

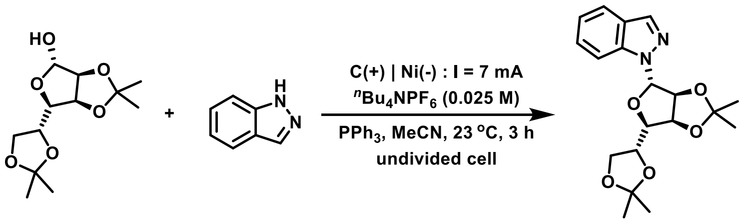
1-((3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)-2*H*-benzo[*d*][1,2,3]triazole (3ae)



The starting (3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3] dioxol-4-ol (104 mg, 0.4 mmol) was reacted with 1*H*-benzo[*d*][1,2,3]triazole (36 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a white solid in 75% yield (81 mg). (α:β = 5:1)

Rf (petroleum ether/ethyl acetate = 3:1): 0.3; ^{1}**H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.03 – 7.81 (m, 2H), 7.48 – 7.39 (m, 2H), 5.46 (s, 1H), 4.87 – 4.82 (m, 1H), 4.67 (d, *J* = 6.0 Hz, 1H), 4.49 – 4.43 (m, 1H), 4.29 – 4.24 (m, 1H), 4.14 – 4.07 (m, 2H), 1.46 (s, 6H), 1.39 (s, 3H), 1.31 (s, 3H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ (ppm): 138.7, 125.9, 114.8, 112.5, 109.0, 101.0, 85.4, 79.9, 79.5, 73.2, 66.4, 26.6, 25.6, 25.0, 24.2. **HRMS m/z (ESI)** calcd for $C_{15}H_{21}N_4O_2$ ($[M+H]^+$) 362.1710, found 362.1712.

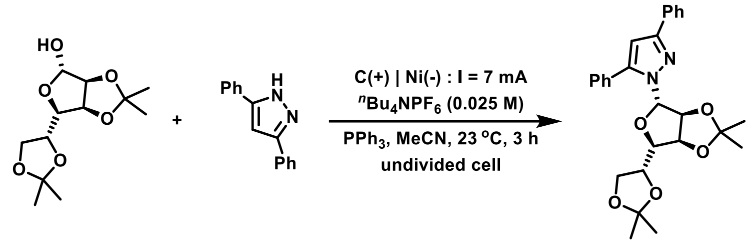
1-((3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)-1*H*-indazole (3af)



The starting (3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3] dioxol-4-ol(105 mg, 0.4 mmol) was reacted with 1*H*-indazole (36 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a white solid in 82% yield (89 mg). (α:β = 10:1)

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 8.09 (s, 1H), 7.79 – 7.75(m, 1H), 7.52 – 7.48 (m, 1H), 7.42 – 7.36 (m, 1H), 7.19 – 7.15 (m, 1H), 5.44 (s, 1H), 4.86 – 4.78 (m, 1H), 4.64 (d, J = 5.9 Hz, 1H), 4.47 – 4.41 (m, 1H), 4.31 – 4.24 (m, 1H), 4.15 – 4.04 (m, 2H), 1.47 (s, 6H), 1.40 (s, 3H), 1.32 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 139.9, 134.4, 126.8, 123.0, 120.9, 120.8, 112.5, 109.7, 108.9, 101.0, 85.4, 79.9, 79.5, 73.3, 66.3, 26.6, 25.7, 25.1, 24.3. **HRMS m/z (ESI)** calcd for $C_{16}H_{22}N_3O_2$ ($[M+H]^+$) 361.1758, found 361.1760.

1-((3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)-3,5-diphenyl-1*H*-pyrazole (3ag)



The starting (3a*S*,4*S*,6*R*,6a*S*)-6-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3] dioxol-4-ol (105 mg, 0.4 mmol) was reacted with 3,5-diphenyl-1*H*-pyrazole (66 mg, 0.30 mmol) and PPh_3 (105 mg, 0.4 mmol) according to General Procedure. Water (10 mL) was added to the reaction mixture and extracted with ethyl acetate (10 mL × 3). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a white solid in 90% yield (125 mg). (α:β = 5:1)

Rf (petroleum ether/ethyl acetate = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 7.73 – 7.68 (m, 4H), 7.38 – 7.28 (m, 6H), 6.81 (s, 1H), 5.42 (s, 1H), 4.81 – 4.77 (m, 1H), 4.62 (d, J = 5.9 Hz, 1H), 4.44 – 4.38 (m, 1H), 4.25 – 4.21 (m, 1H), 4.10 – 4.00 (m, 2H), 1.46 (s, 3H), 1.41 (s, 3H), 1.36 (s, 3H), 1.30 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm): 148.7, 131.0, 128.8, 128.2, 125.6, 112.5, 109.0, 101.2, 100.2, 85.4, 80.1, 79.6, 73.2, 66.4, 26.7, 25.7, 25.1, 24.3. **HRMS m/z (ESI)** calcd for $C_{24}H_{28}N_3O_2$ ($[M+H]^+$) 463.2227, found 463.2226.

4. Procedure for cyclic voltammetry (CV)

Cyclic voltammetry was performed in a three-electrode cell under air at room temperature. A steady glassy carbon disk electrode (3 mm in diameter) was used as the working electrode; a platinum plate was used as the counter electrode; the reference was an Ag/AgNO$_3$ electrode with (0.01 M) AgNO$_3$ in acetonitrile. 8 mL acetonitrile solvent containing (0.025 M) ^{n}Bu$_4$NPF$_6$ was used as the blank. The spectrums were recorded with the scan rate of 50 mV s^{-1}, from 0 V to 3.0 V (starting from 0 V). The CV of PPh$_3$, Boc-pyrrolidin-2-ol, and 4-nitro-1*H*-pyrazole were conducted respectively.

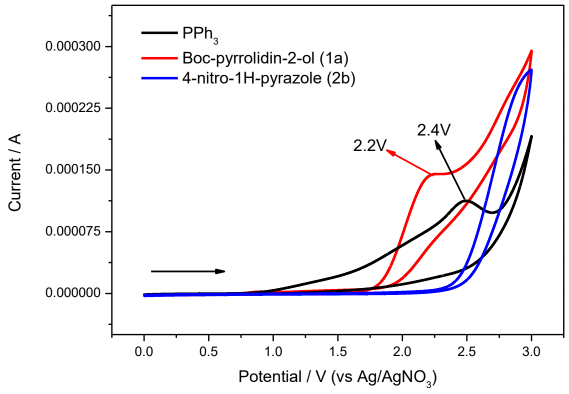
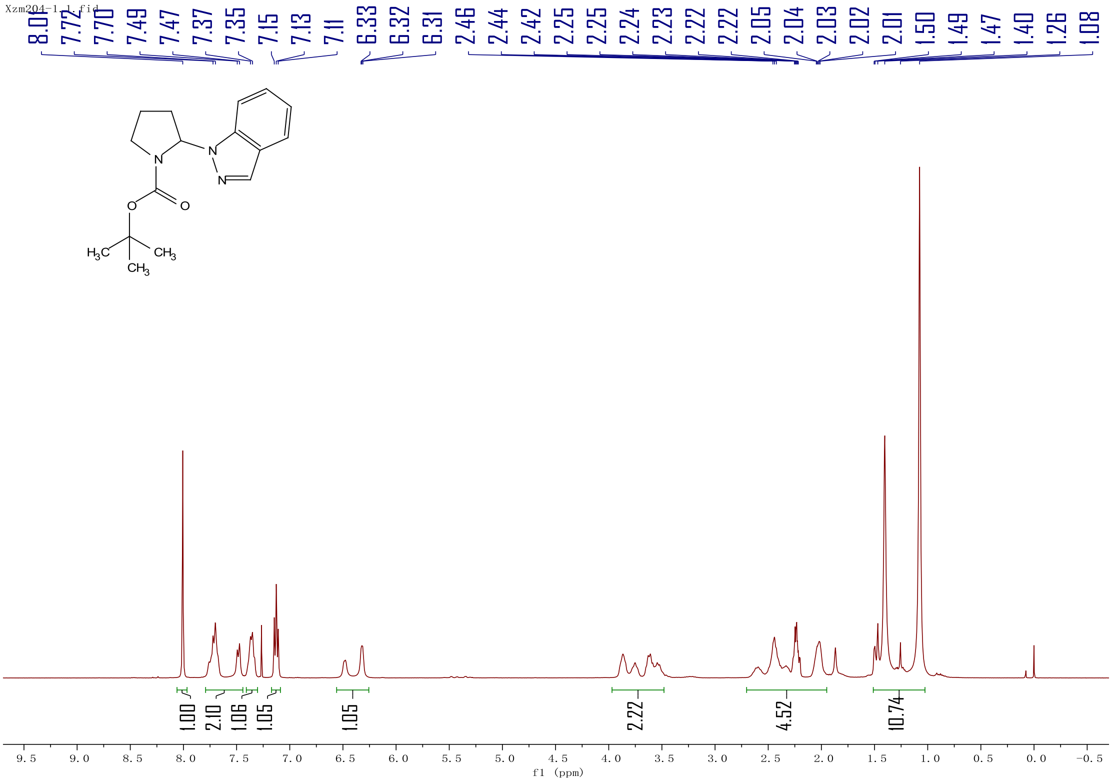
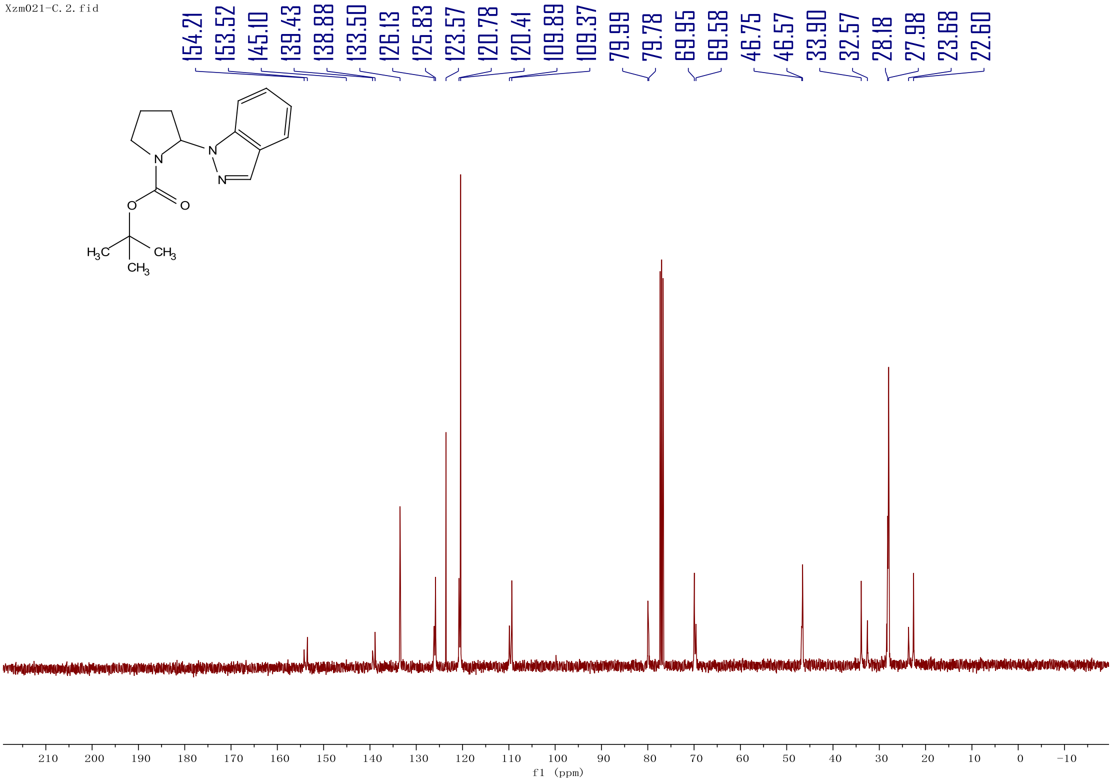


Fig 1. Cyclic voltammograms recorded in (0.025 M) ^{n}Bu$_4$NPF$_6$-MeCN solution: scan rate: 50 mV s^{-1}; starting potential: 0 V; glass carbon (3 mm diameter, Working Electrode); platinum plate (Counter Electrode); Ag/AgNO$_3$ (0.01 M) AgNO$_3$ in MeCN (Reference Electrode); Concentrations: PPh$_3$ (0.2 mmol / 8 ml MeCN), Boc-pyrrolidin-2-ol (0.2 mmol / 8 ml MeCN) , 4-nitro-1*H*-pyrazole (0.15 mmol / 8 ml MeCN)

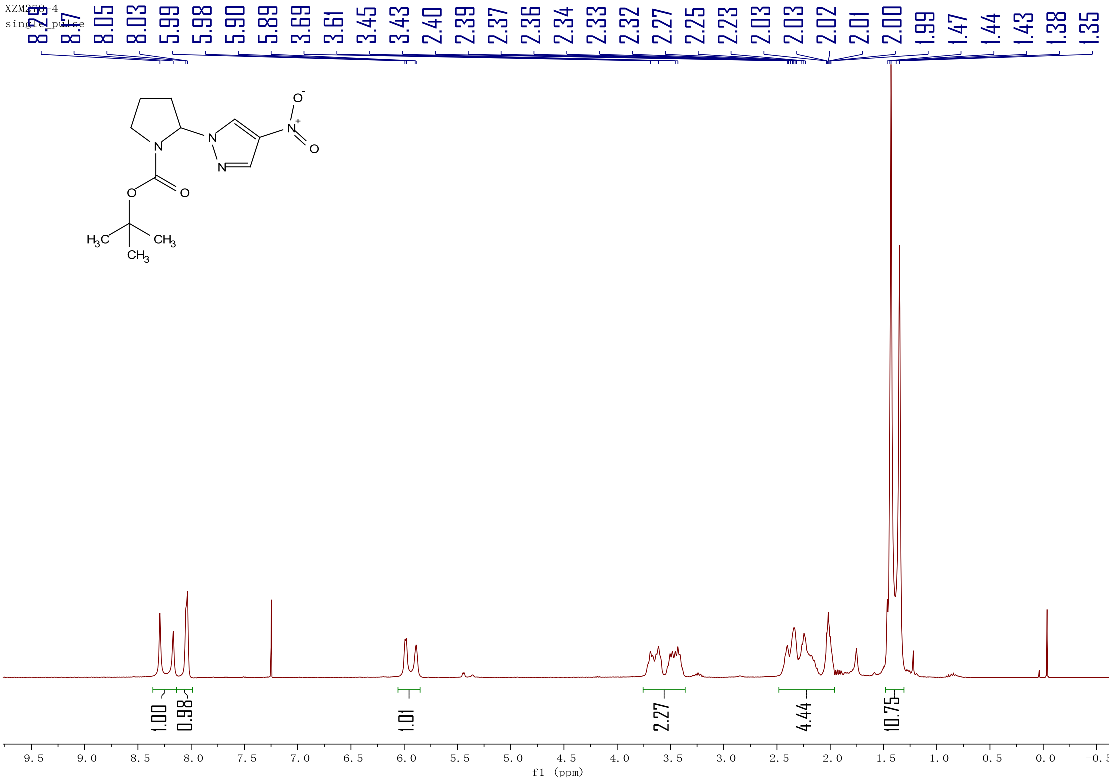
5. ^{1}H and ^{13}C NMR spectra

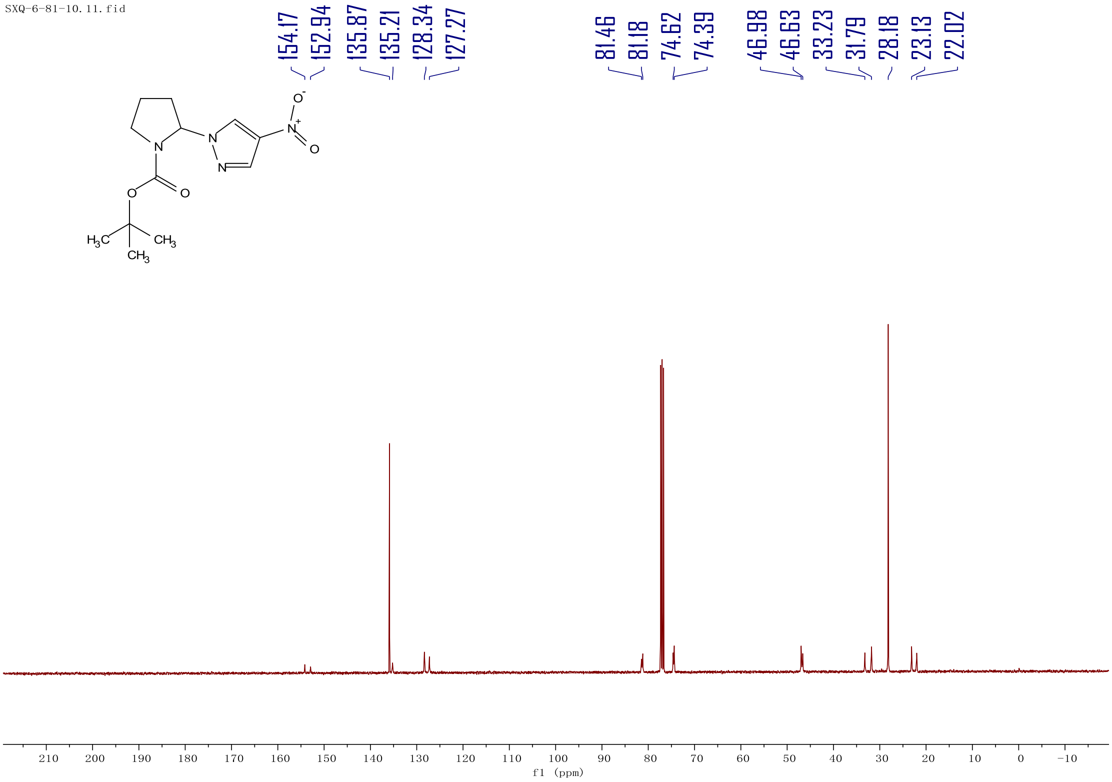
3a (rotameric mixture)



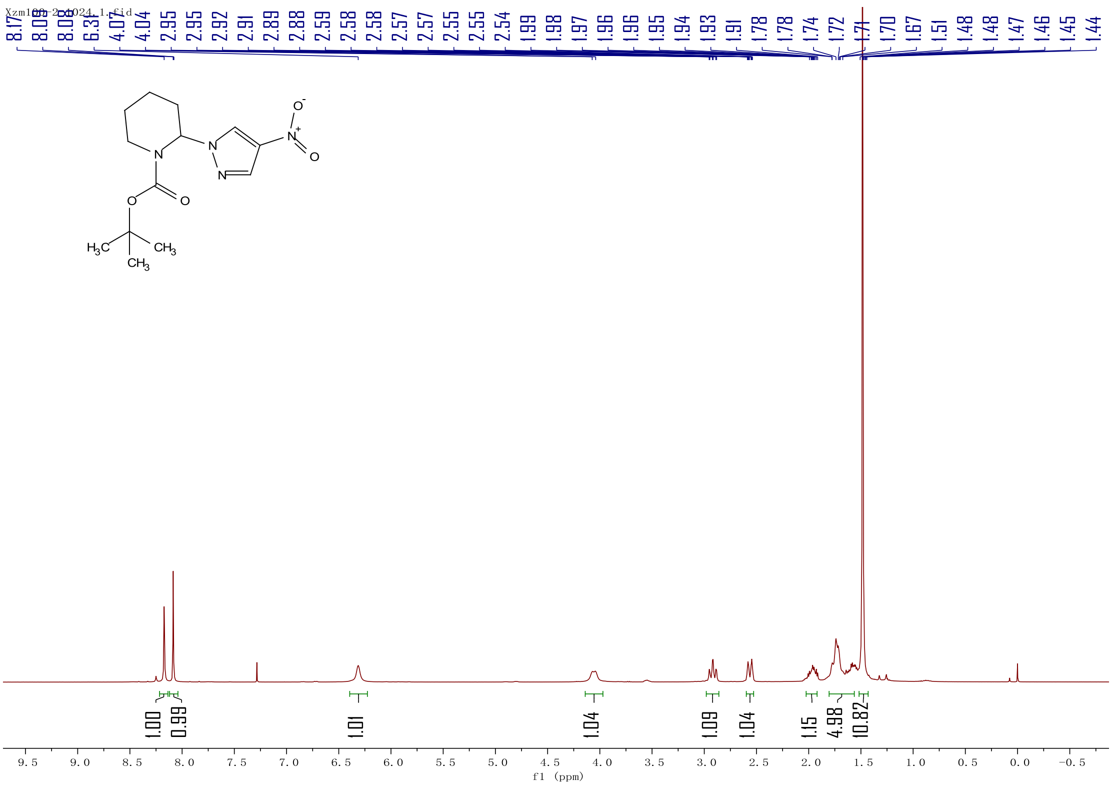


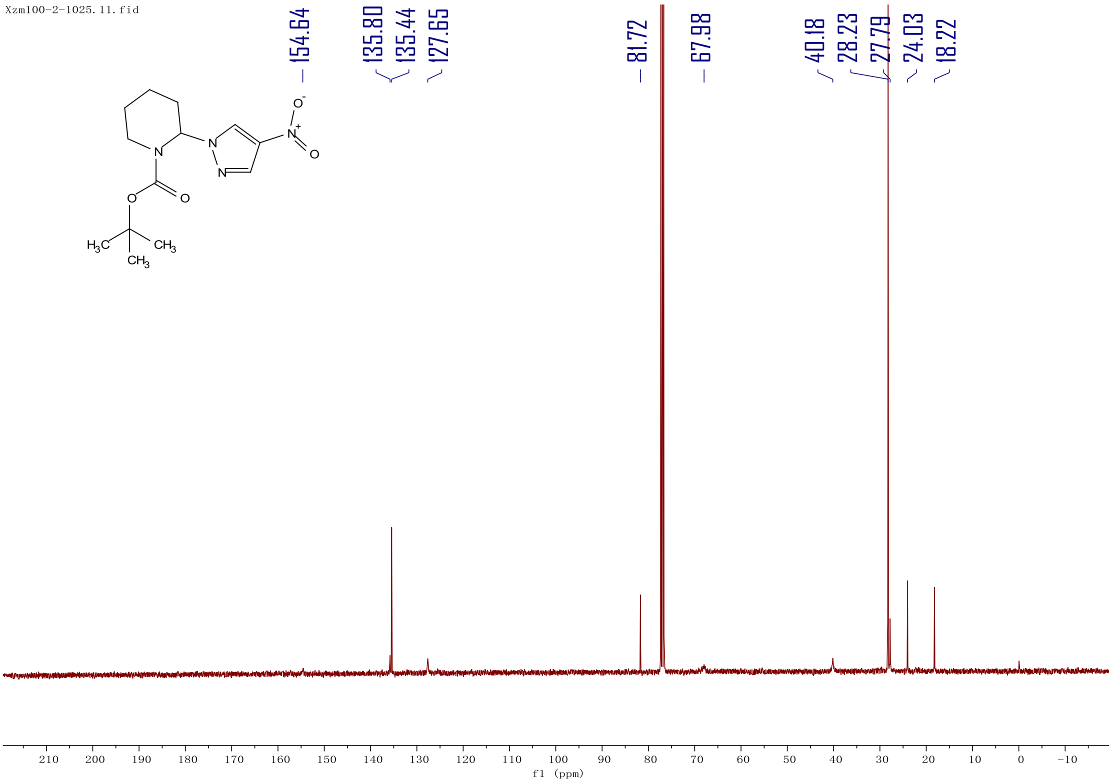
3b (rotameric mixture)



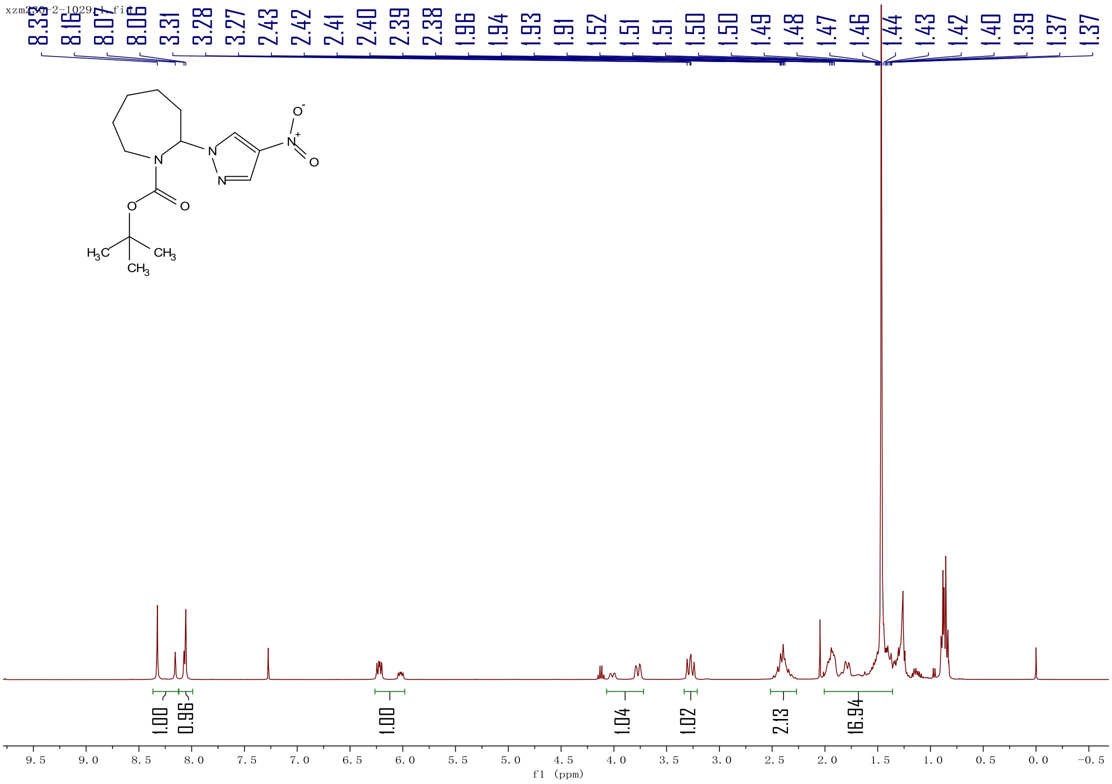


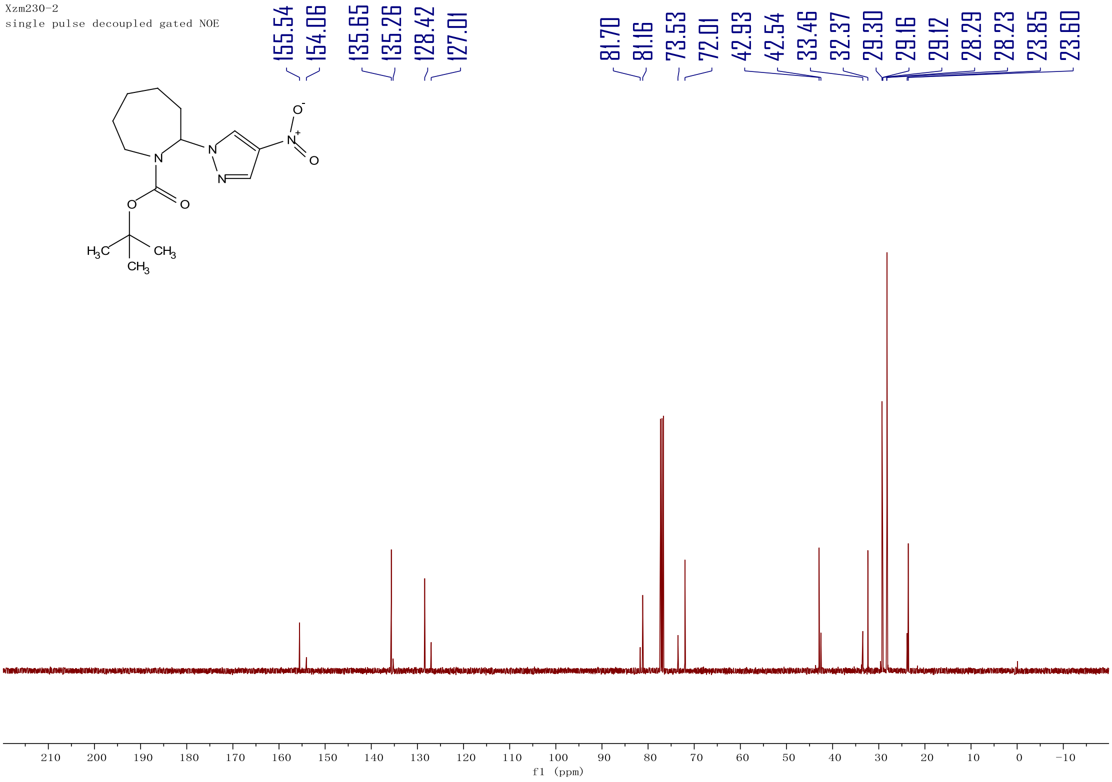
3c


Xzm100-2-1024.1.fid
8.17
8.09
8.08
6.31
4.07
4.04
2.95
2.95
2.92
2.91
2.89
2.88
2.59
2.58
2.58
2.57
2.57
2.55
2.55
2.54
1.99
1.98
1.97
1.96
1.96
1.95
1.94
1.93
1.91
1.78
1.78
1.74
1.72
1.71
1.70
1.67
1.51
1.48
1.48
1.47
1.46
1.45
1.44
O-
N+
O
N
N
N
O
O
H3C
CH3
CH3
1.00
0.99
1.01
1.04
1.09
1.04
1.15
4.98
10.82
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

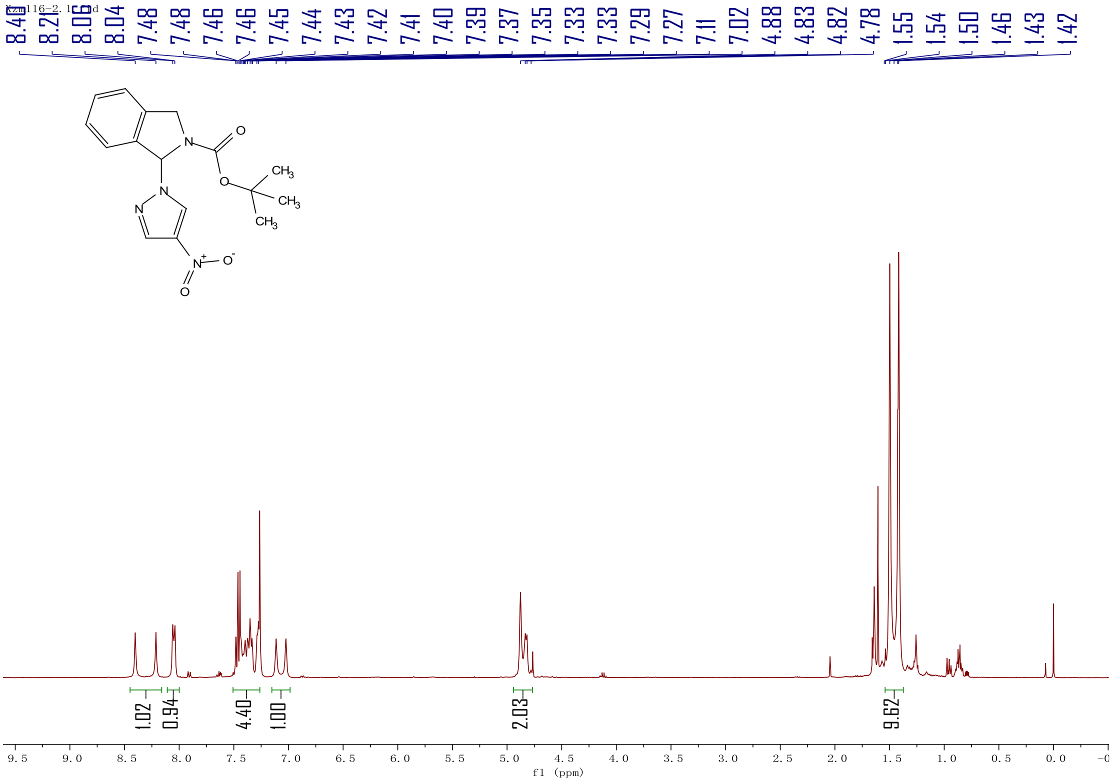

Xzm100-2-1025.11.fid
154.64
135.80
135.44
127.65
81.72
67.98
40.18
28.23
27.79
24.03
18.22
O-
N+
O
N
N
N
O
O
H3C
CH3
CH3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

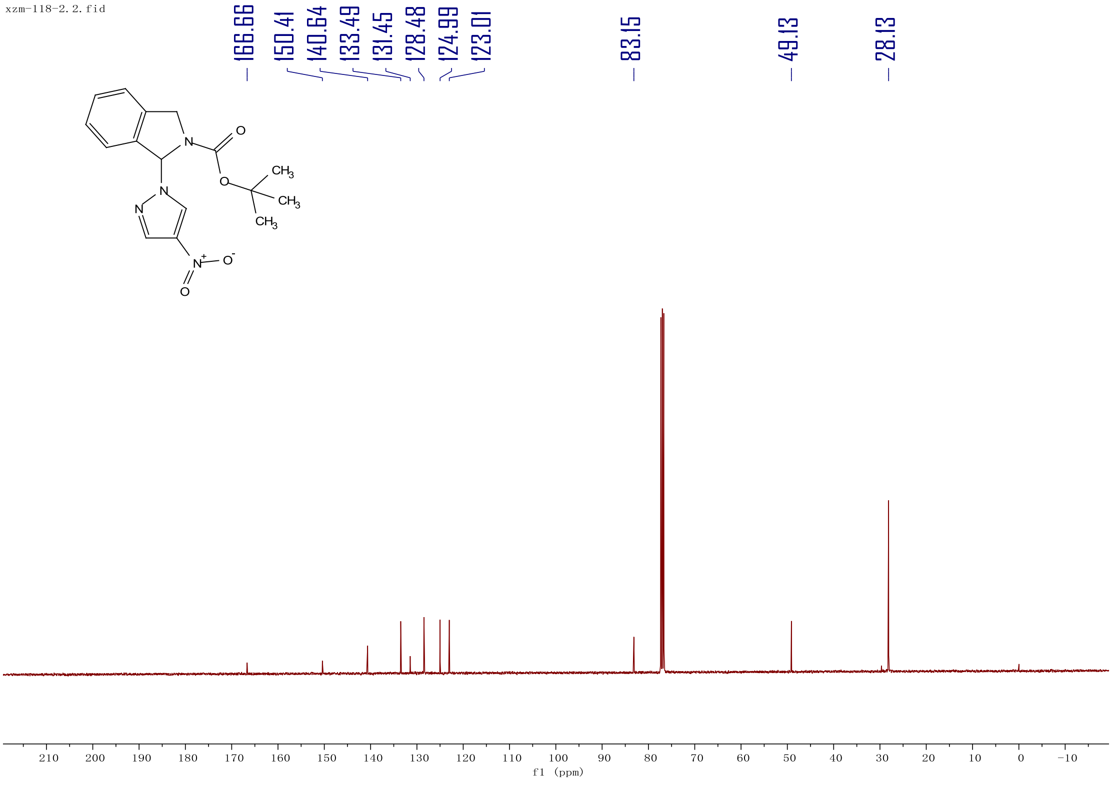
3d (rotameric mixture)



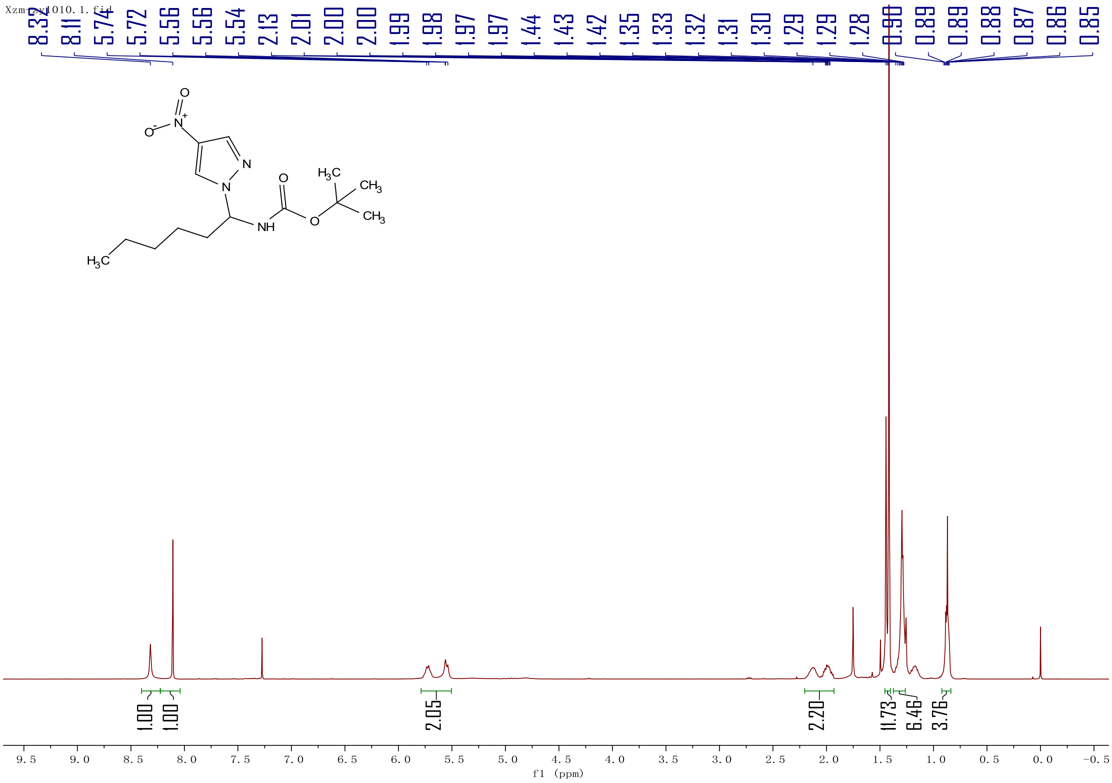


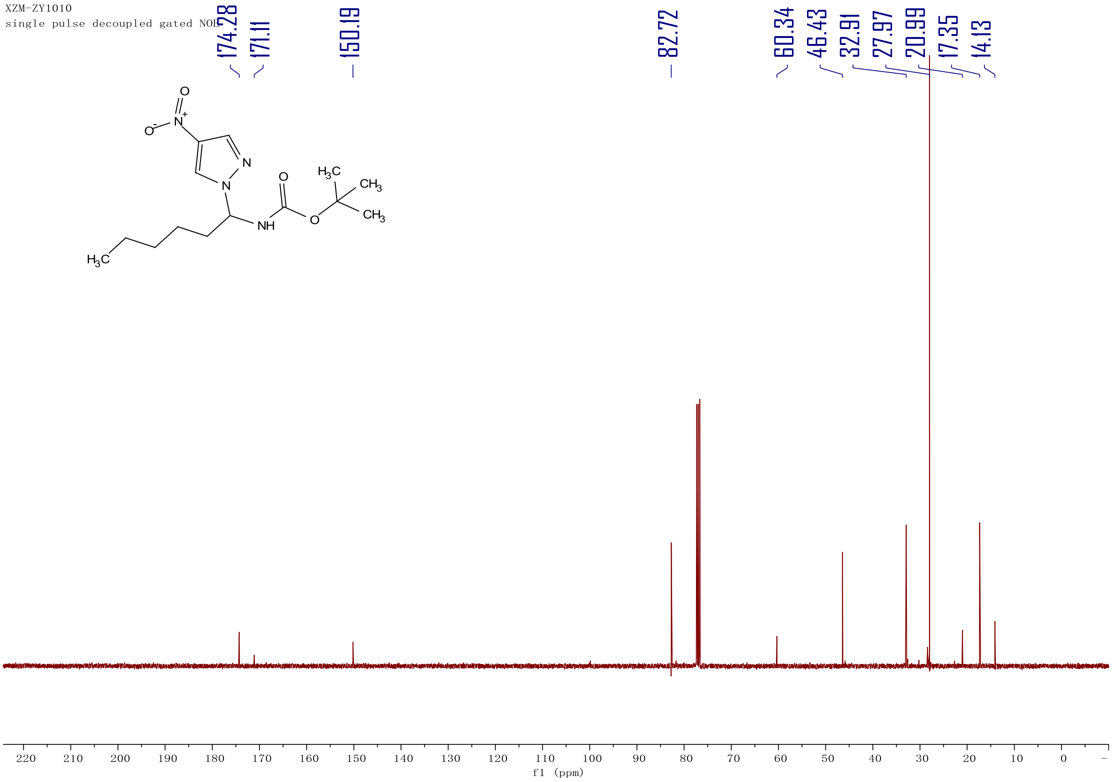
3e (rotameric mixture)



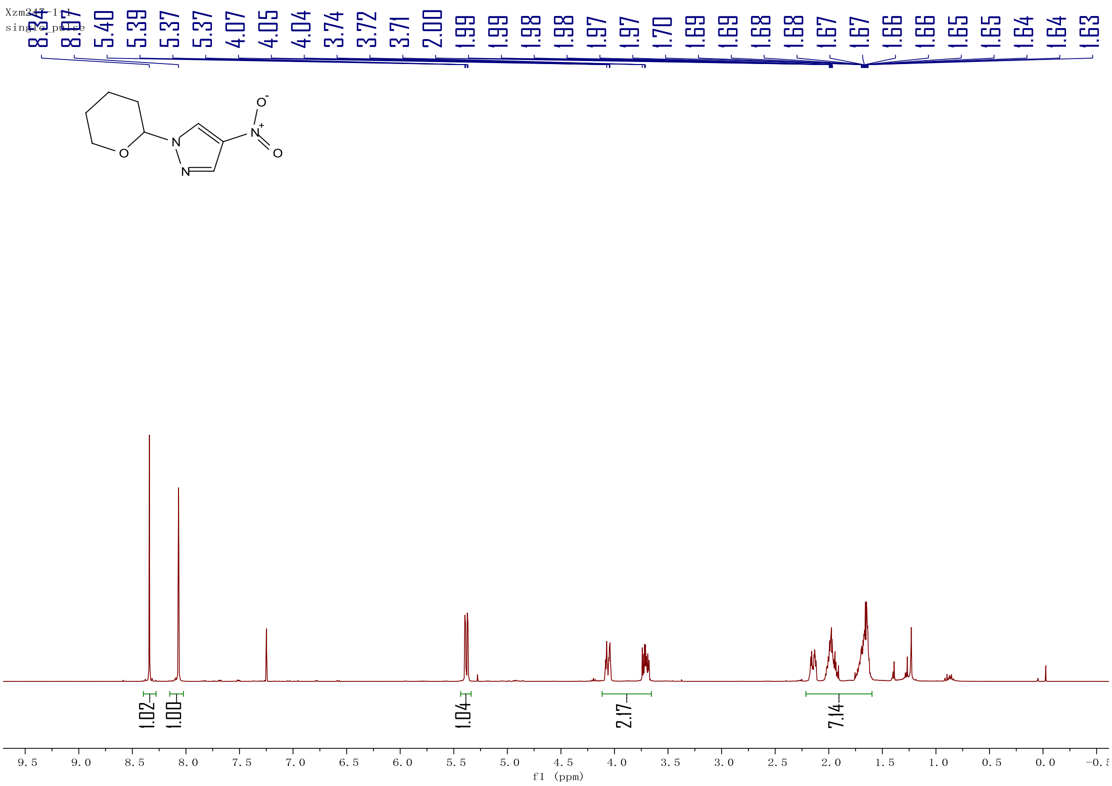


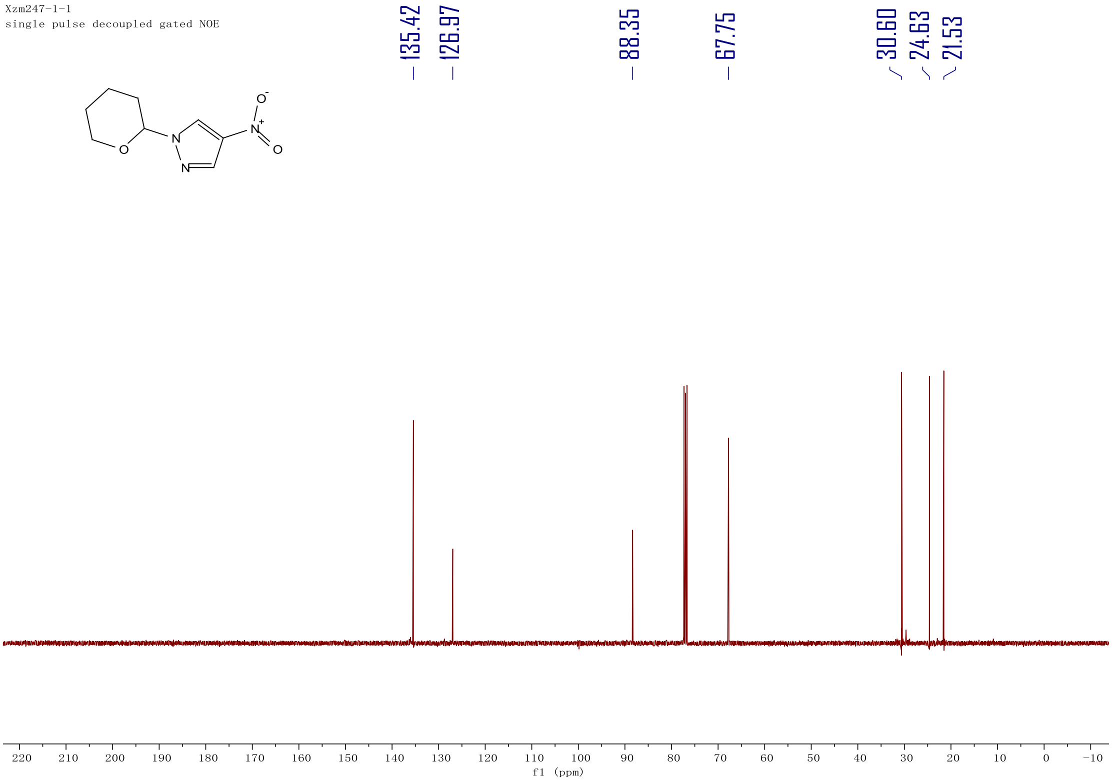
3f


Xzm-zy1010.1.fid
8.32
8.11
5.74
5.72
5.56
5.56
5.54
2.13
2.01
2.00
2.00
1.99
1.98
1.97
1.97
1.44
1.43
1.42
1.35
1.33
1.32
1.31
1.30
1.29
1.29
1.28
0.90
0.89
0.89
0.88
0.87
0.86
0.85
O
N
O
N
N
H3C
CH3
CH3
O
NH
O
H3C
1.00
1.00
2.05
2.20
11.73
6.46
3.76
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

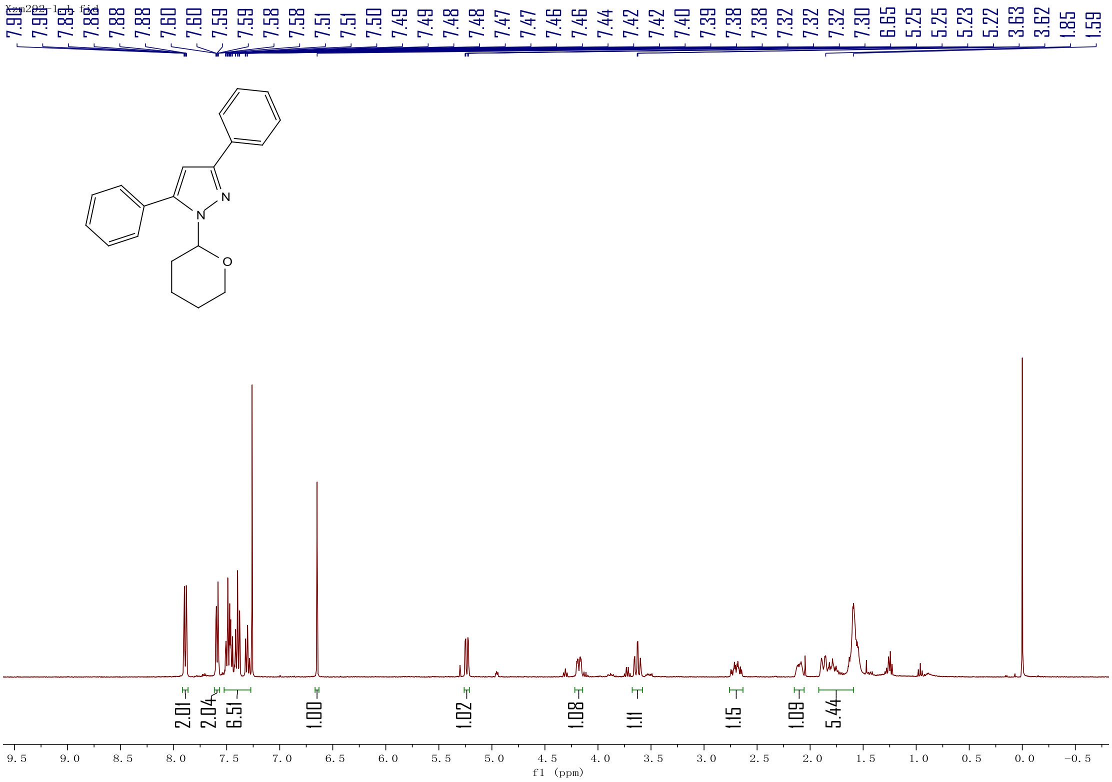

XZM-ZY1010
single pulse decoupled gated NOE
174.28
171.11
150.19
82.72
60.34
46.43
32.91
27.97
20.99
17.35
14.13
O
N
O
N
N
H3C
CH3
CH3
O
NH
O
H3C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

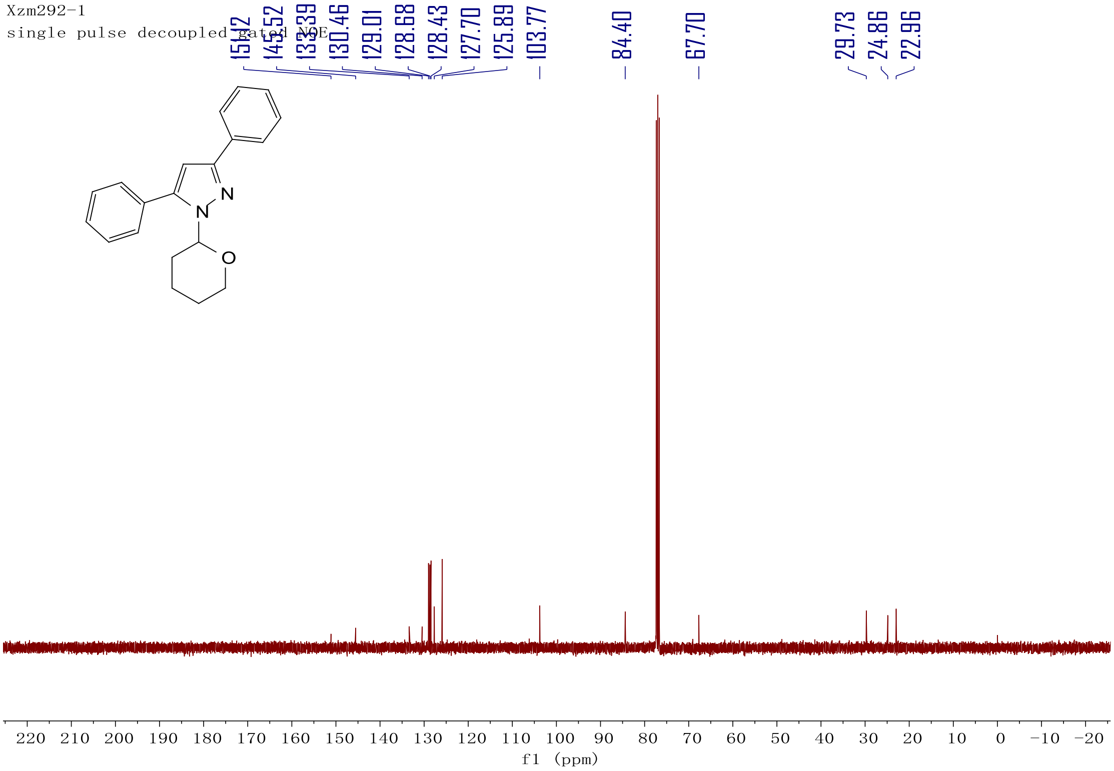
3g


Xzm247-1-1
single pulse
8.34 8.07 5.40 5.39 5.37 5.37 4.07 4.05 4.04 3.74 3.72 3.71 2.00 1.99 1.99 1.98 1.98 1.97 1.97 1.70 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.63
O
N
N
N
O
O
1.02
1.00
1.04
2.17
7.14
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

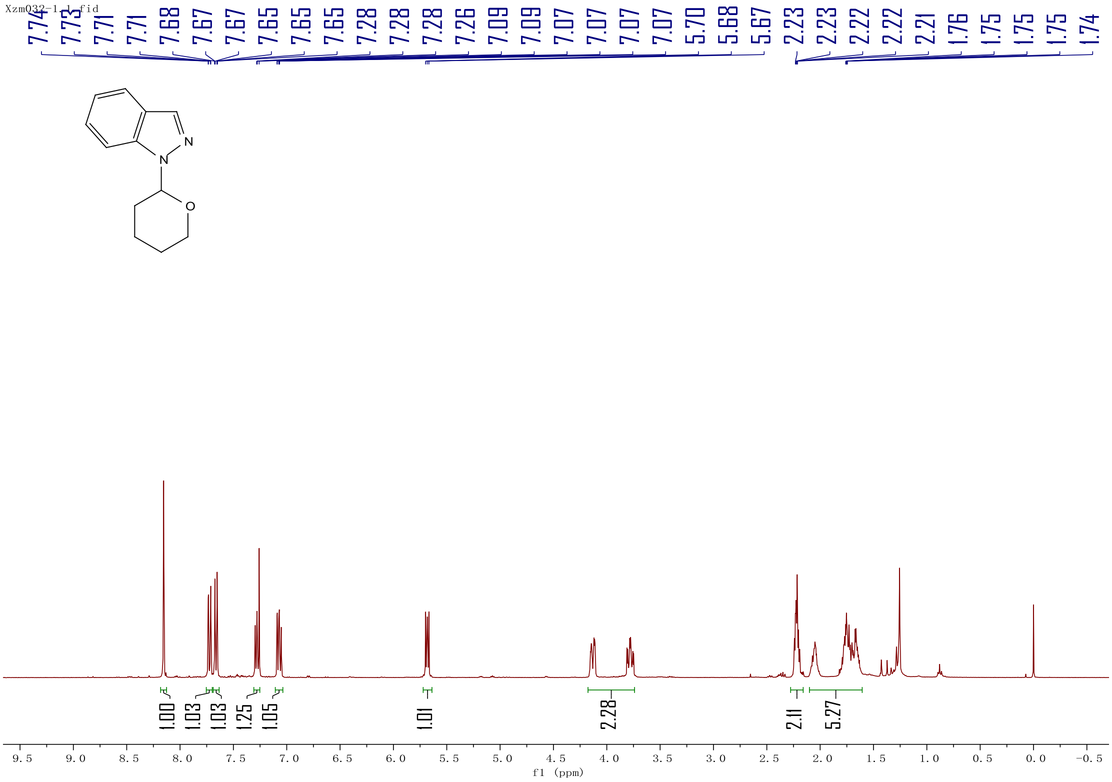

Xzm247-1-1
single pulse decoupled gated NOE
135.42
126.97
88.35
67.75
30.60
24.63
21.53
O
N
N
N
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

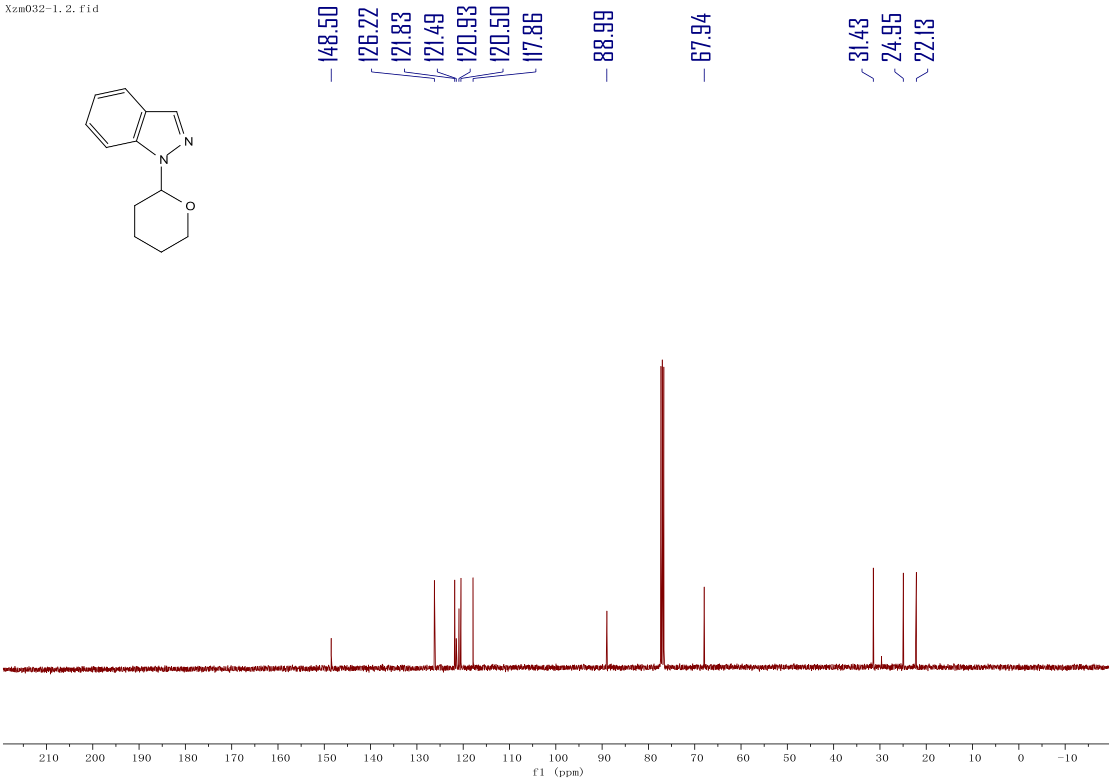
3h


Xzm292-1-1.fid
7.90
7.90
7.89
7.88
7.88
7.88
7.60
7.60
7.59
7.59
7.58
7.58
7.51
7.51
7.50
7.49
7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.44
7.42
7.42
7.40
7.39
7.38
7.38
7.32
7.32
7.32
7.30
6.65
5.25
5.25
5.23
5.22
3.63
3.62
1.85
1.59
N
N
O
2.01
2.04
6.51
1.00
1.02
1.08
1.11
1.15
1.09
5.44
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

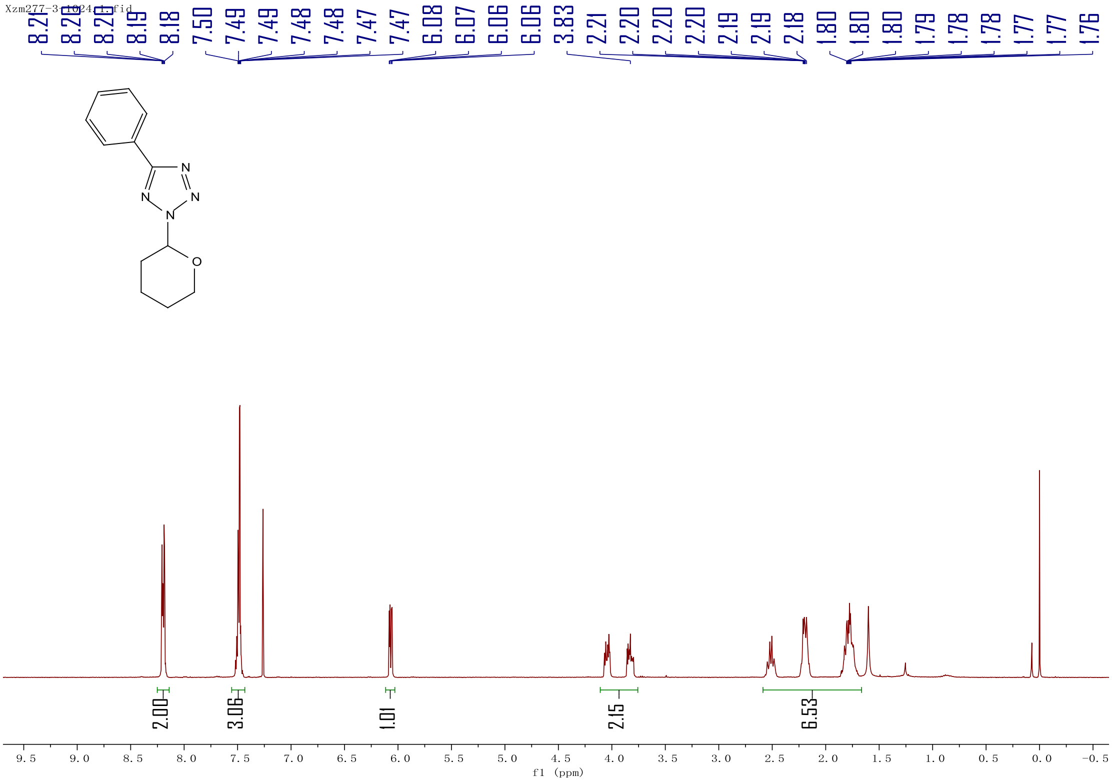

Xzm292-1
single pulse decoupled gated NOE
151.12
145.52
133.39
130.46
129.01
128.68
128.43
127.70
125.89
103.77
84.40
67.70
29.73
24.86
22.96
N
N
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

3i

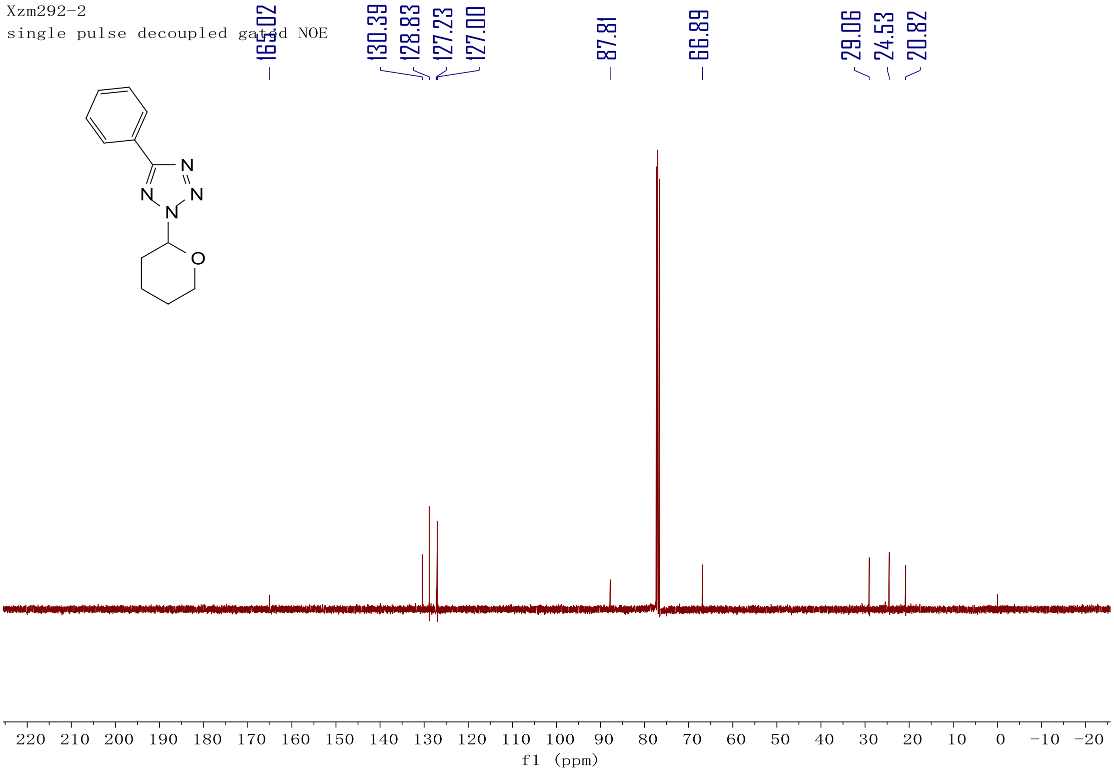

Xzm032-1.1.fid
7.74
7.73
7.71
7.71
7.68
7.67
7.67
7.65
7.65
7.65
7.28
7.28
7.28
7.26
7.09
7.09
7.07
7.07
7.07
7.07
5.70
5.68
5.67
2.23
2.23
2.22
2.22
2.21
1.76
1.75
1.75
1.75
1.74
N
N
O
1.00
1.03
1.03
1.25
1.05
1.01
2.28
2.11
5.27
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


Xzm032-1.2.fid
148.50
126.22
121.83
121.49
120.93
120.50
117.86
88.99
67.94
31.43
24.95
22.13
N
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3j

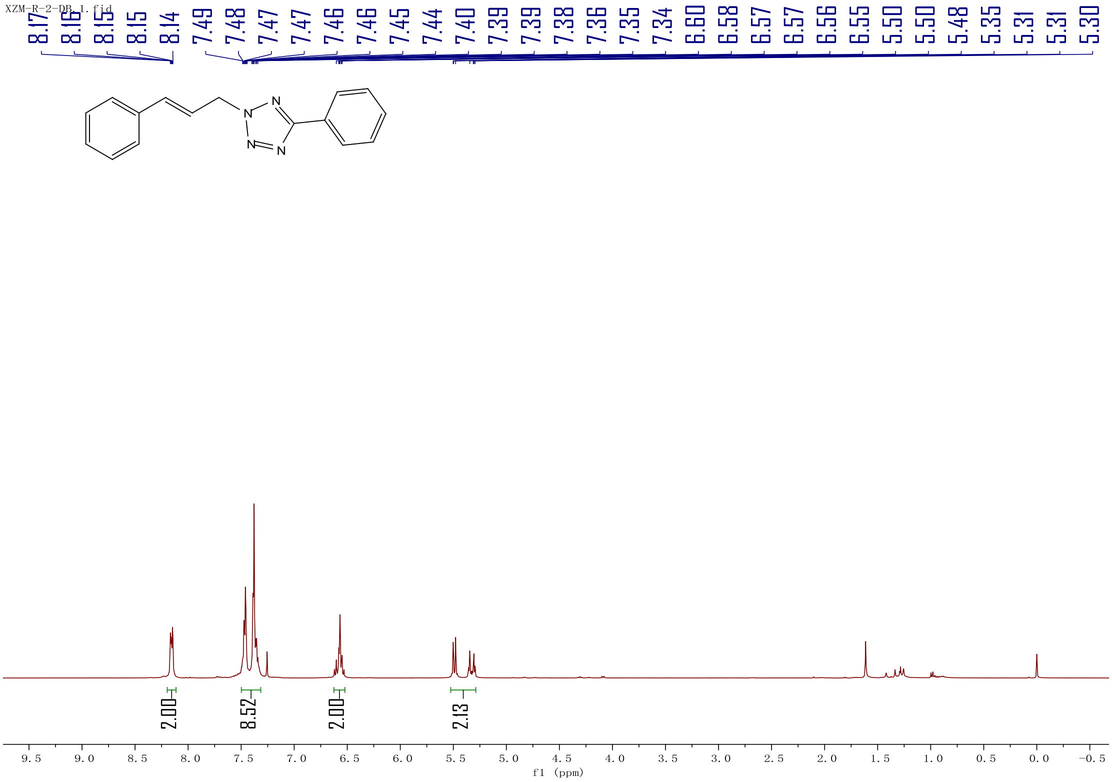


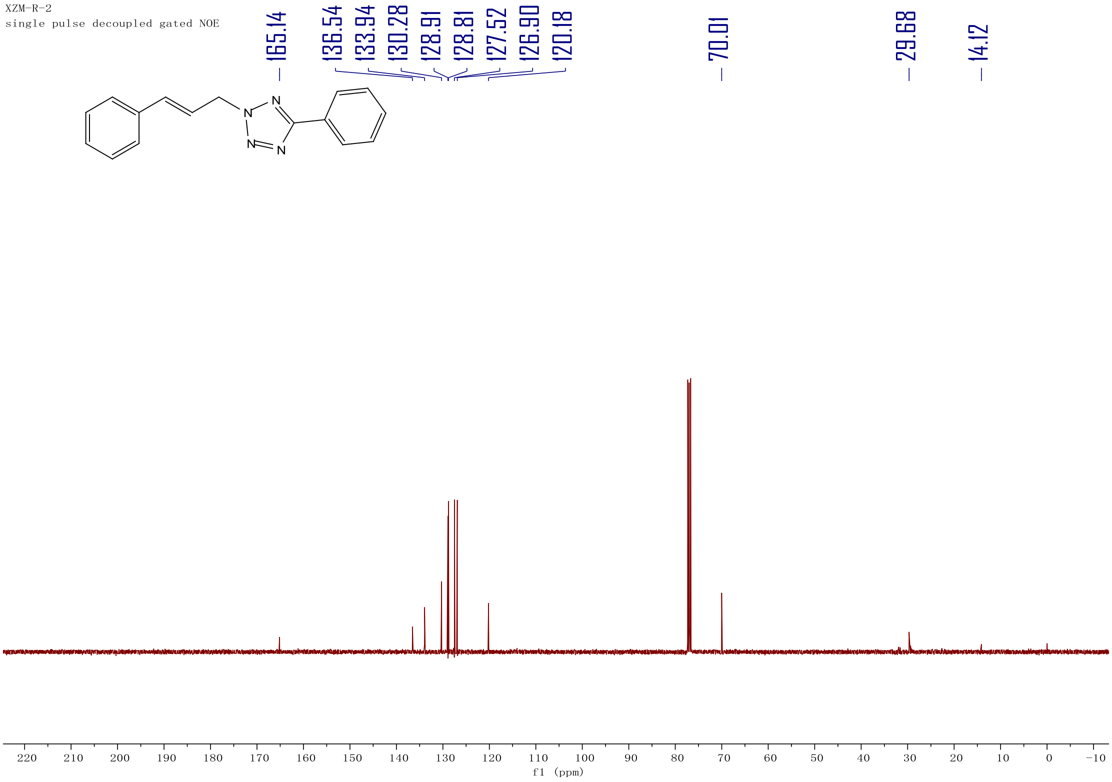
Xzm277-3-1024-1.fid
8.21
8.20
8.20
8.19
8.18
7.50
7.49
7.49
7.48
7.48
7.47
7.47
6.08
6.07
6.06
6.06
3.83
2.21
2.20
2.20
2.20
2.19
2.19
2.18
1.80
1.80
1.80
1.79
1.78
1.78
1.78
1.77
1.77
1.76
N
N
N
N
O
2.00
3.06
1.01
2.15
6.53
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)



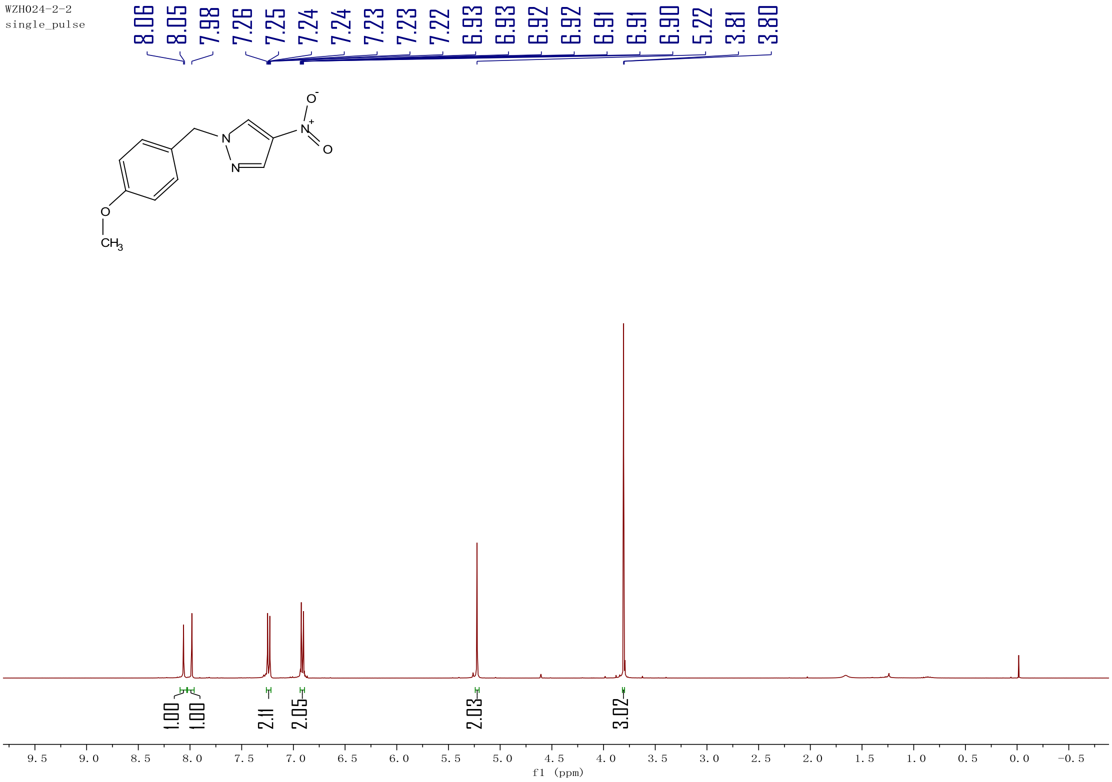
Xzm292-2
single pulse decoupled gated NOE
165.02
130.39
128.83
127.23
127.00
87.81
66.89
29.06
24.53
20.82
N
N
N
N
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

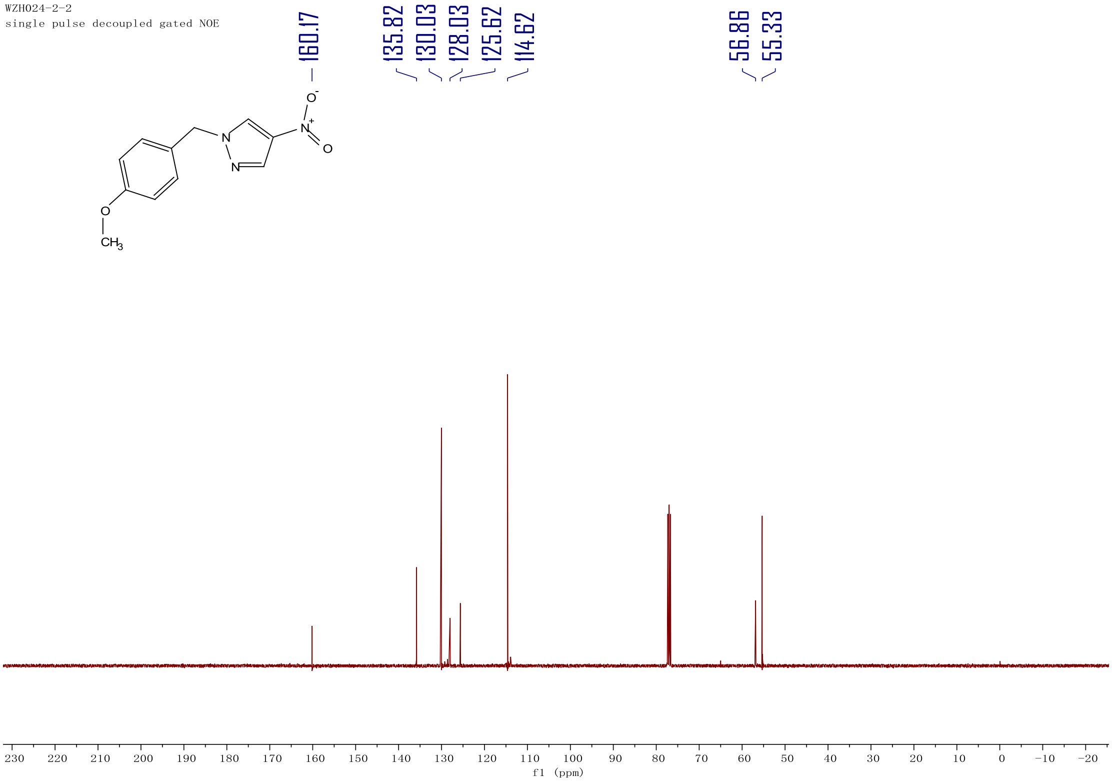
3k


XZM-R-2-DB_1.fid
8.17
8.16
8.15
8.15
8.14
7.49
7.48
7.47
7.47
7.46
7.46
7.45
7.44
7.40
7.39
7.39
7.38
7.36
7.35
7.34
6.60
6.58
6.57
6.57
6.56
6.55
5.50
5.50
5.48
5.35
5.31
5.31
5.30
N
N
N
N
2.00
8.52
2.00
2.13
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

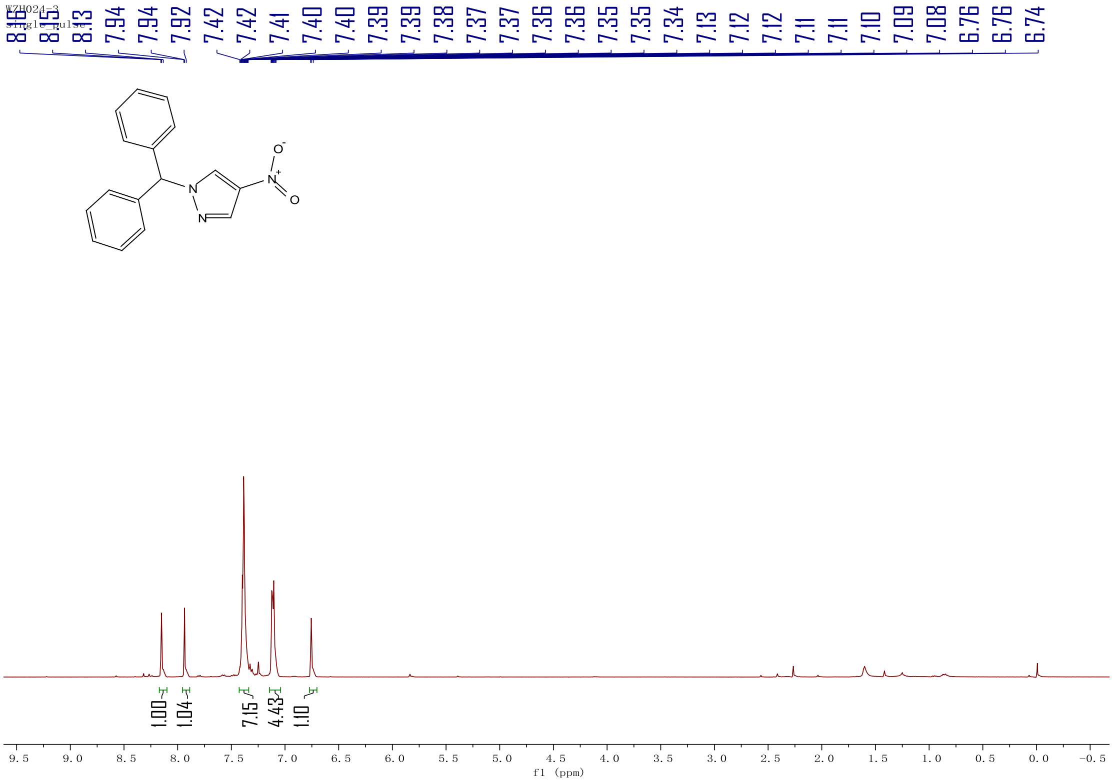

XZM-R-2
single pulse decoupled gated NOE
165.14
136.54
133.94
130.28
128.91
128.81
127.52
126.90
120.18
70.01
29.68
14.12
N
N
N
N
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3l

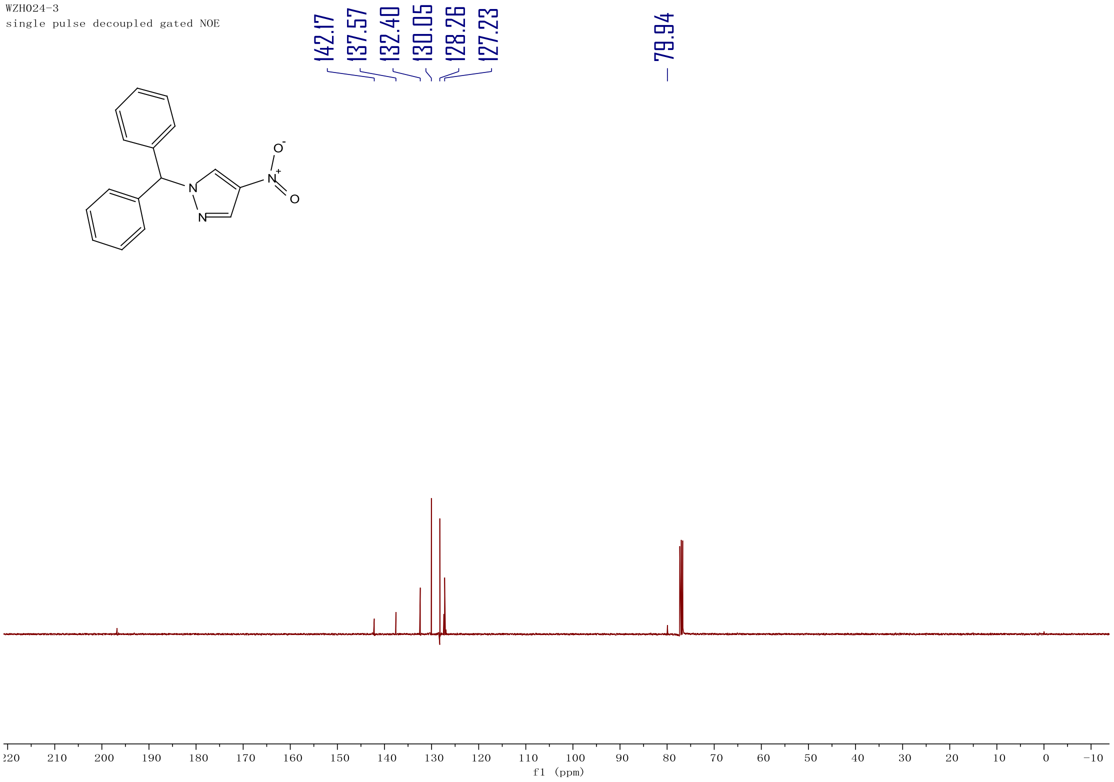

WZH024-2-2
single_pulse
8.06
8.05
7.98
7.26
7.25
7.24
7.24
7.23
7.23
7.22
6.93
6.93
6.92
6.92
6.91
6.91
6.90
5.22
3.81
3.80
O
N
N
N
+
O
O
CH3
1.00
1.00
2.11
2.05
2.03
3.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


WZH024-2-2
single pulse decoupled gated NOE
160.17
135.82
130.03
128.03
125.62
114.62
56.86
55.33
O
N
N
N
+
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

3m

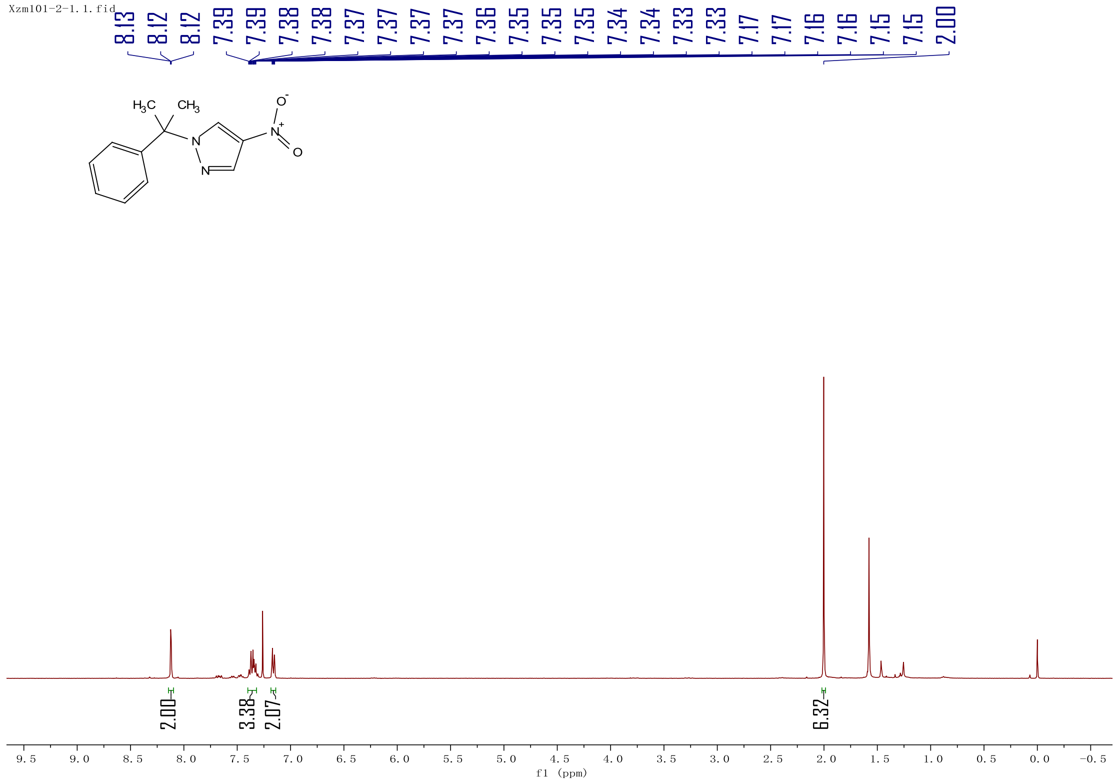


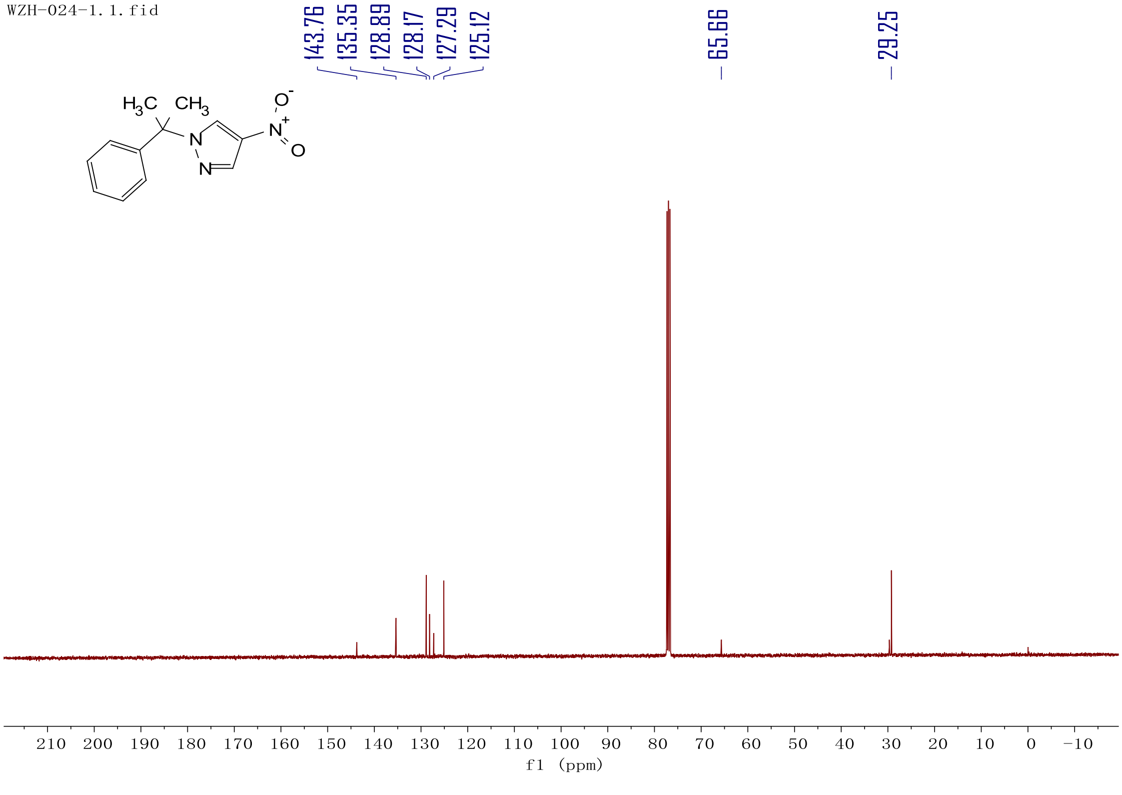
WZH024-3
single pulse
8.16
8.15
8.13
7.94
7.94
7.92
7.42
7.42
7.41
7.40
7.40
7.39
7.39
7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.13
7.12
7.12
7.11
7.11
7.10
7.09
7.08
6.76
6.76
6.74
N
N
N+
O-
O
1.00
1.04
7.15
4.43
1.10
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)



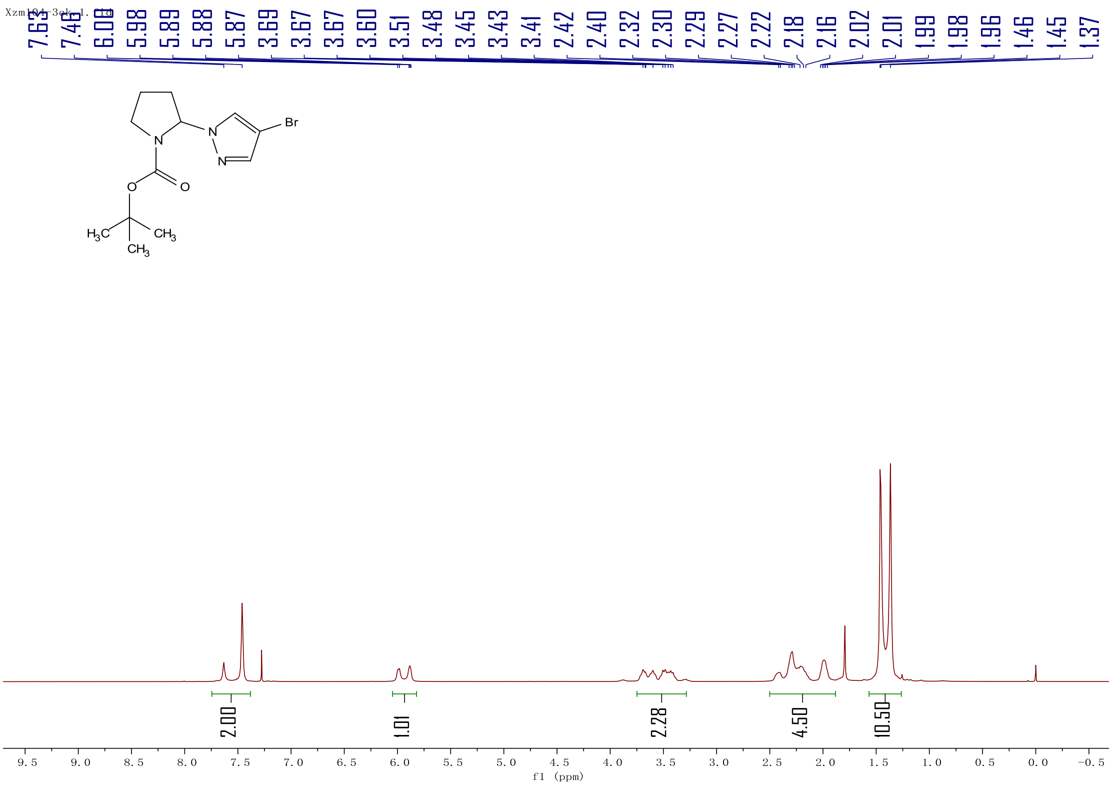
WZH024-3
single pulse decoupled gated NOE
142.17
137.57
132.40
130.05
128.26
127.23
79.94
N
N
N+
O-
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

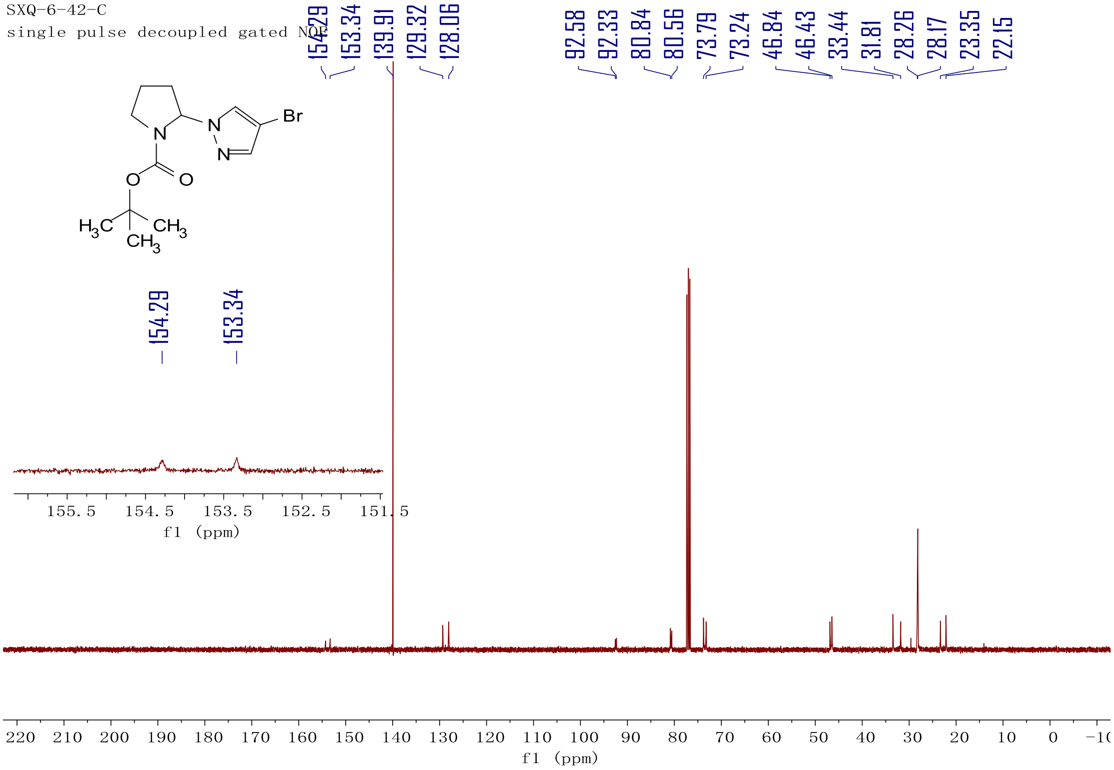
3n


Xzm101-2-1.1.fid
8.13
8.12
8.12
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.37
7.36
7.35
7.35
7.35
7.34
7.34
7.33
7.33
7.17
7.17
7.16
7.16
7.15
7.15
2.00
H3C
CH3
N
N
N+
O-
O
2.00
3.38
2.07
6.32
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

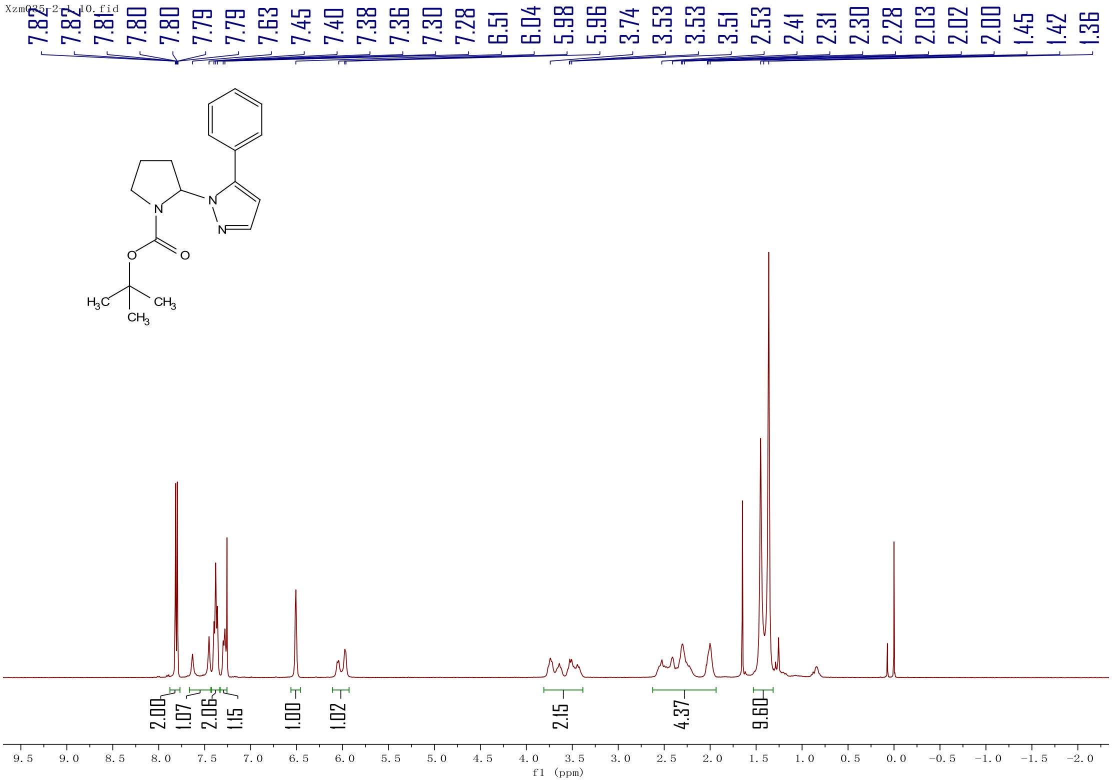

WZH-024-1.1.fid
143.76
135.35
128.89
128.17
127.29
125.12
65.66
29.25
H3C
CH3
N
N
N+
O-
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

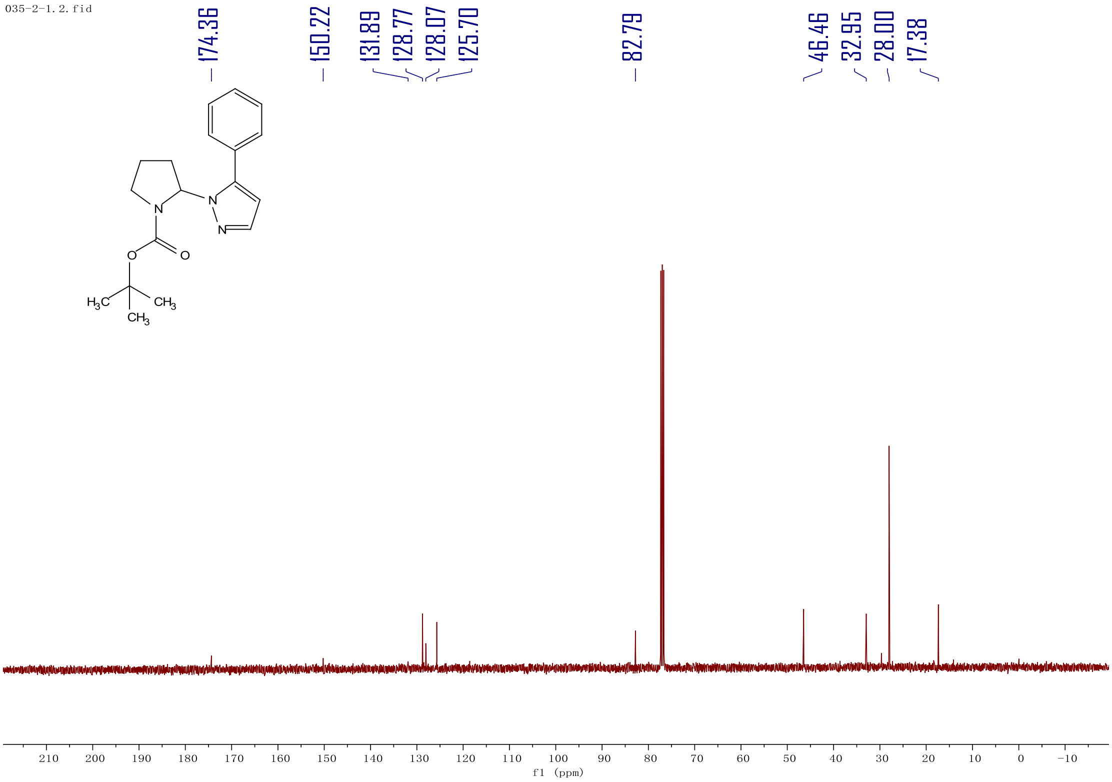
3o (rotameric mixture)



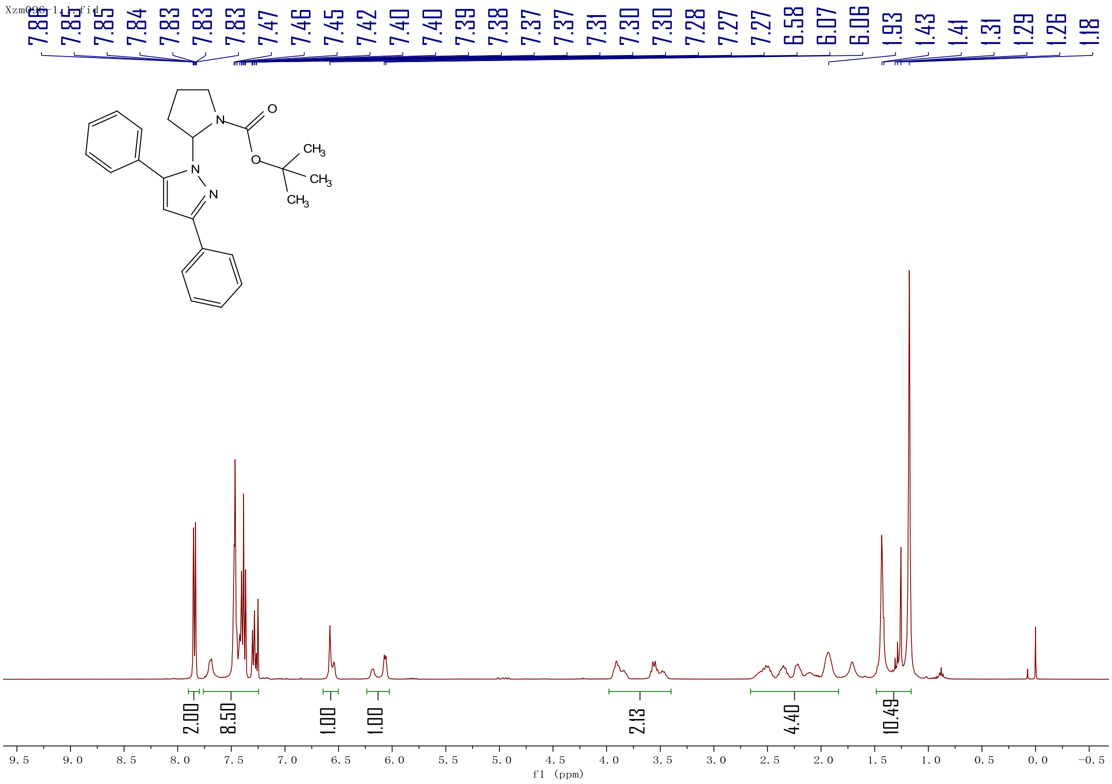


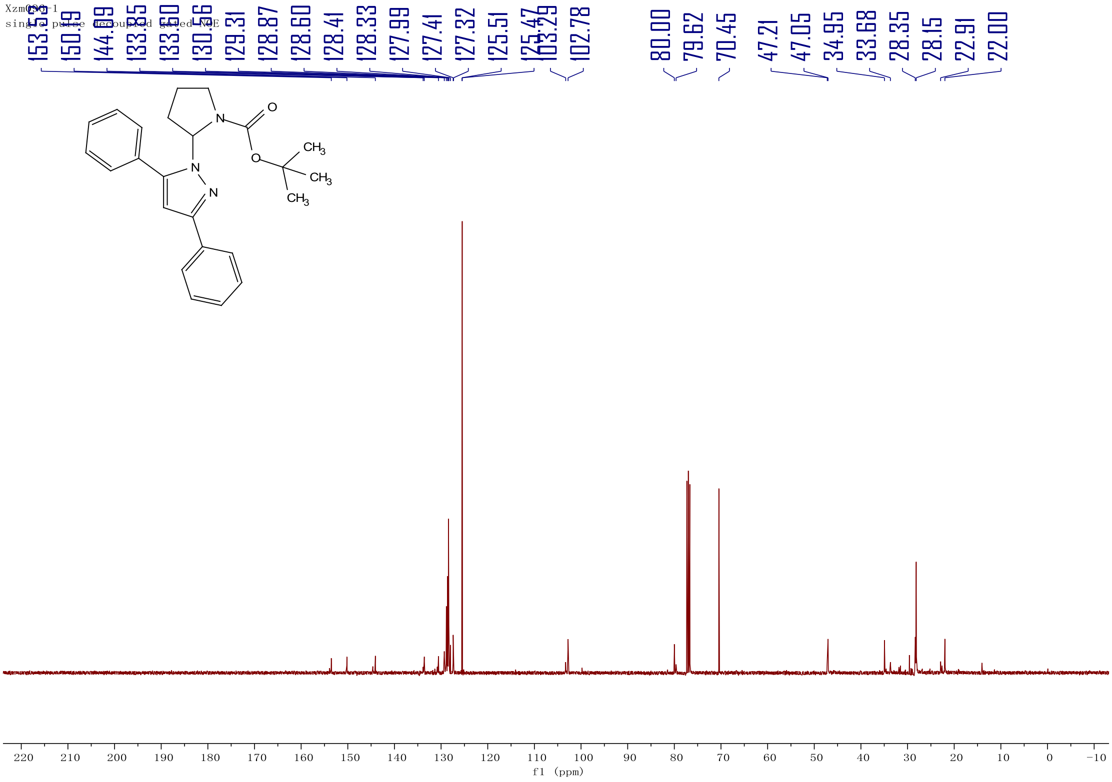
3p


Xzm035-2-1.10.fid
7.82
7.82
7.81
7.80
7.80
7.79
7.79
7.63
7.45
7.40
7.38
7.36
7.30
7.28
6.51
6.04
5.98
5.96
3.74
3.53
3.53
3.51
2.53
2.41
2.31
2.30
2.28
2.03
2.02
2.00
1.45
1.42
1.36
N
N
N
O
O
H_3C
CH_3
CH_3
2.00
1.07
2.06
1.15
1.00
1.02
2.15
4.37
9.60
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0
f1 (ppm)

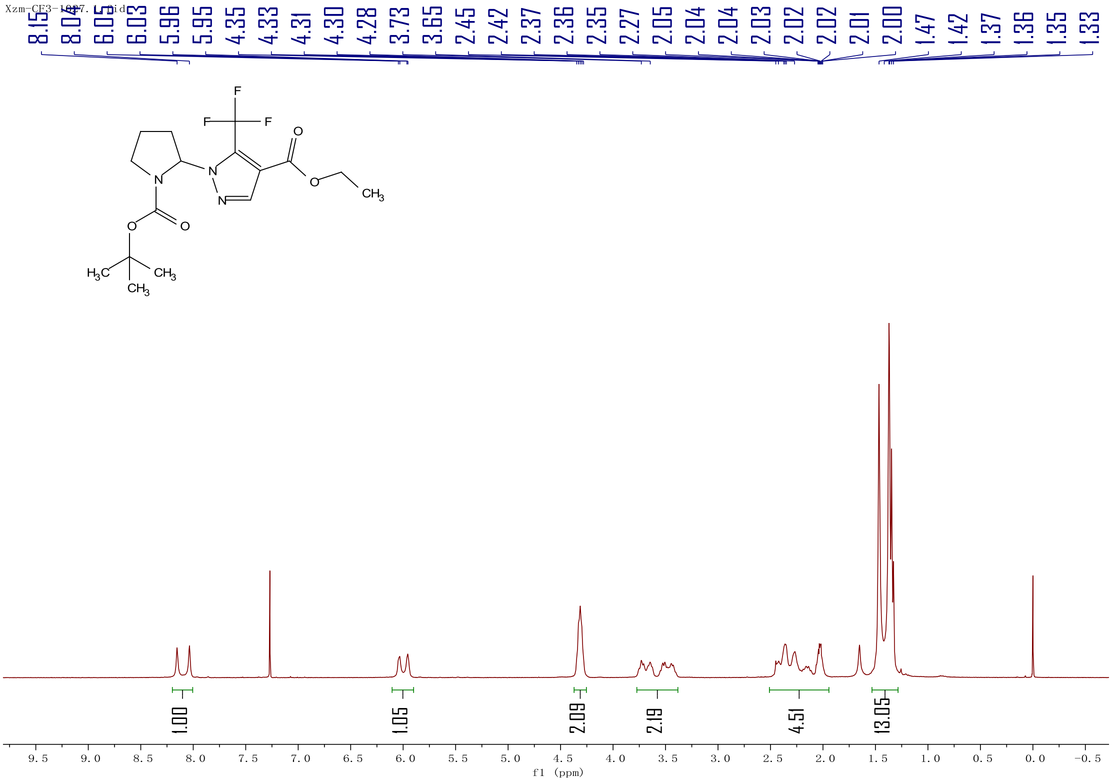

035-2-1.2.fid
174.36
150.22
131.89
128.77
128.07
125.70
82.79
46.46
32.95
28.00
17.38
N
N
N
O
O
H_3C
CH_3
CH_3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

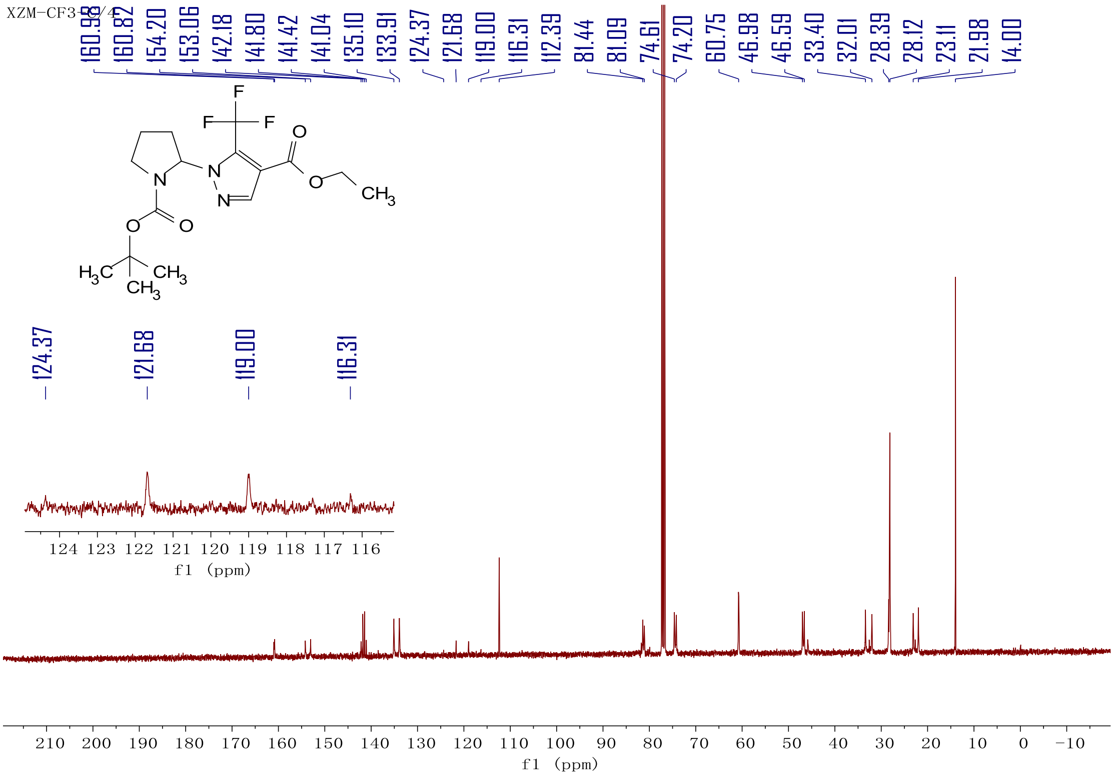
3q (rotameric mixture)



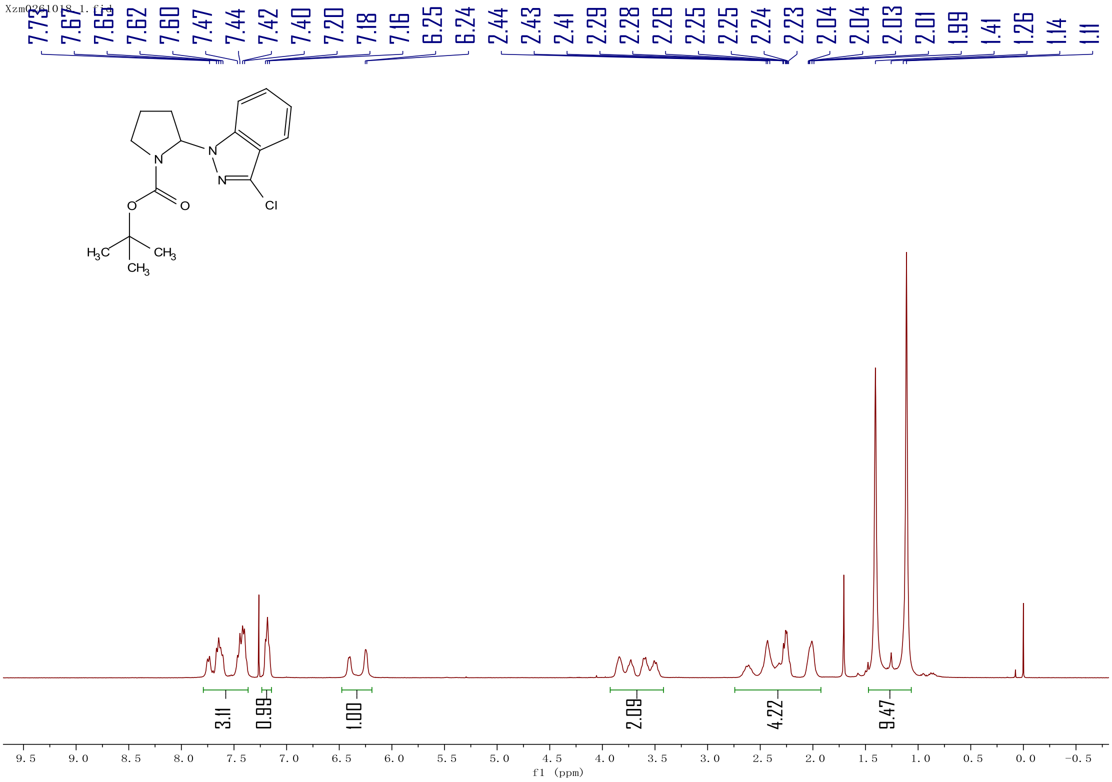


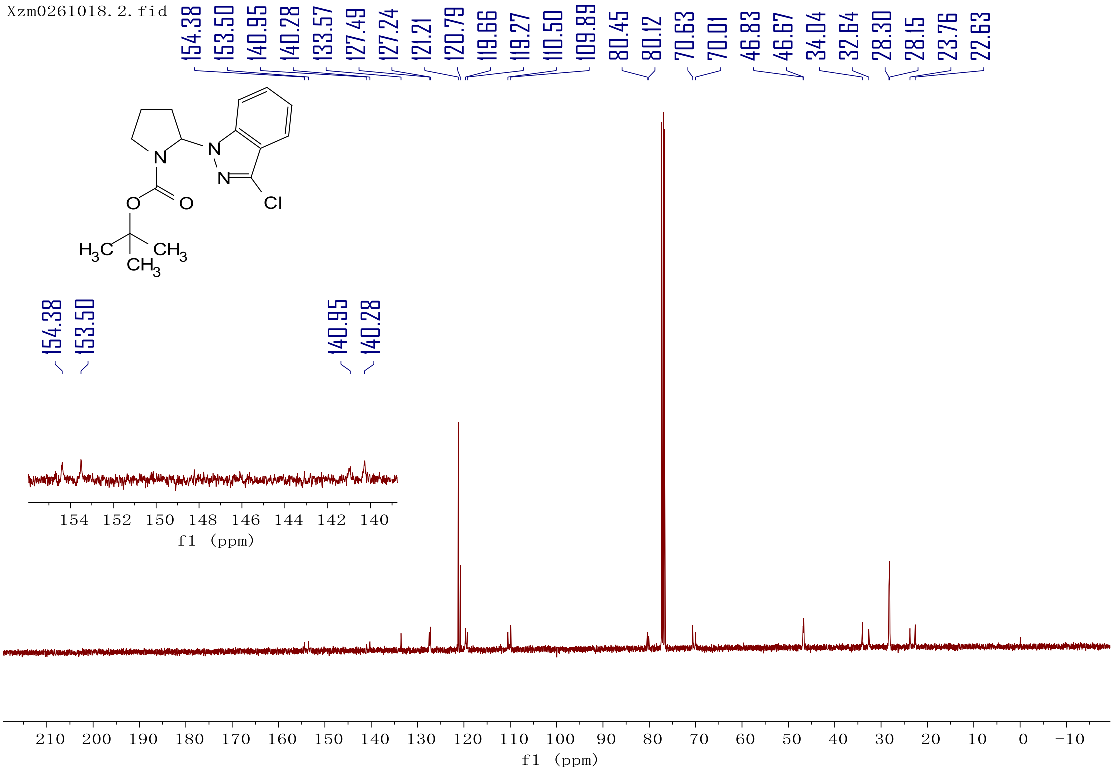
3r (rotameric mixture)



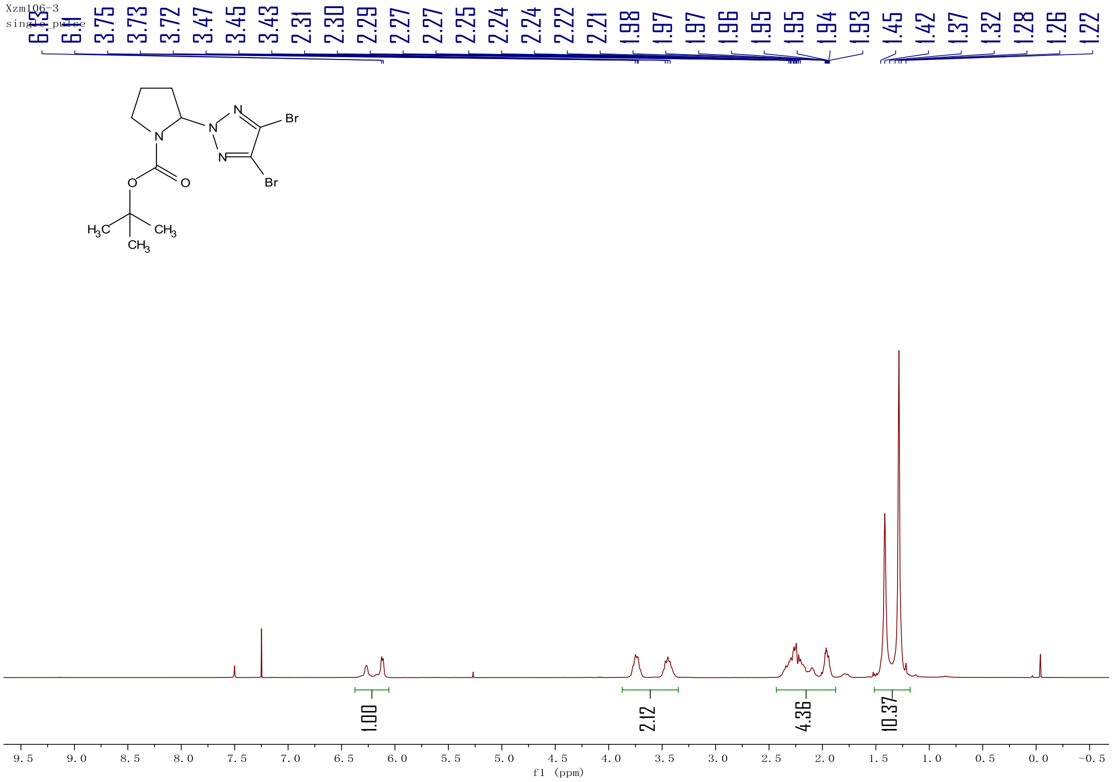


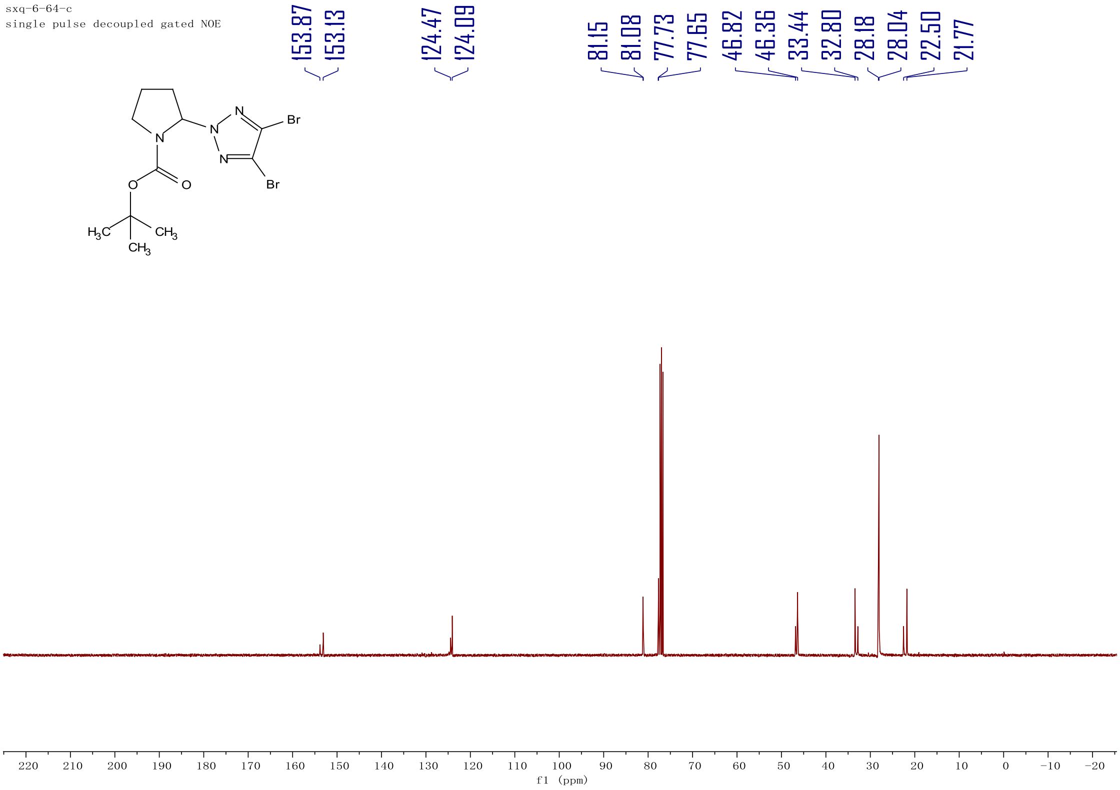
3s (rotameric mixture)



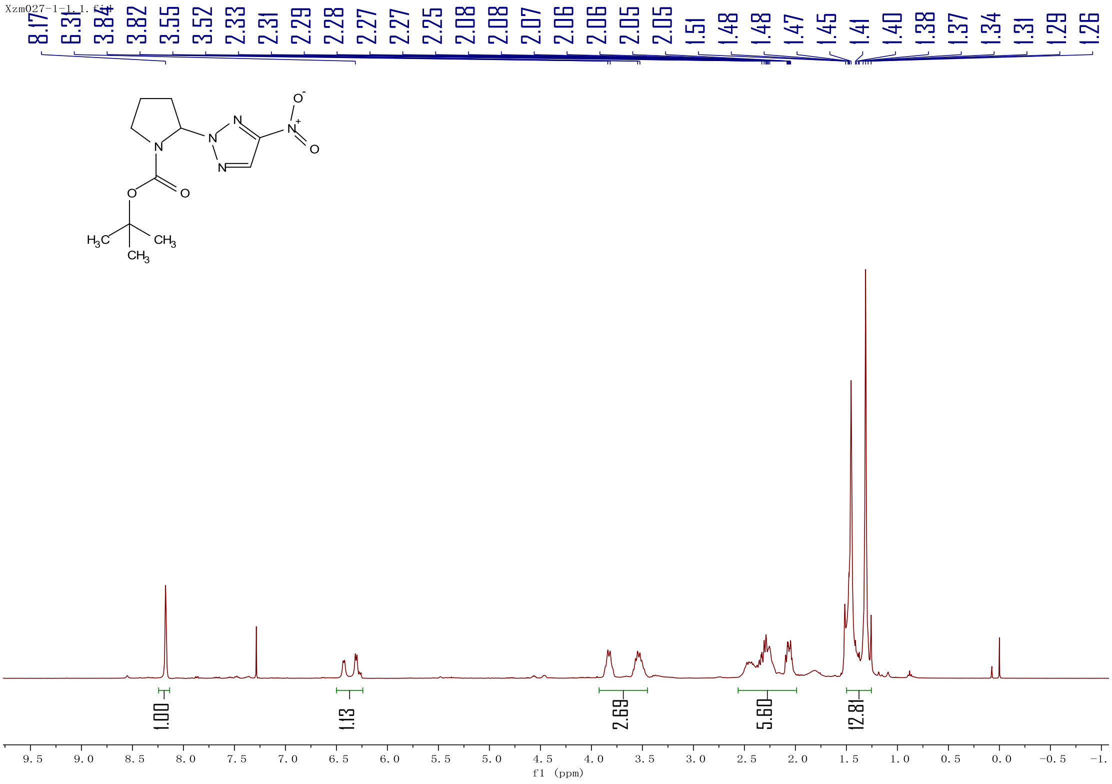


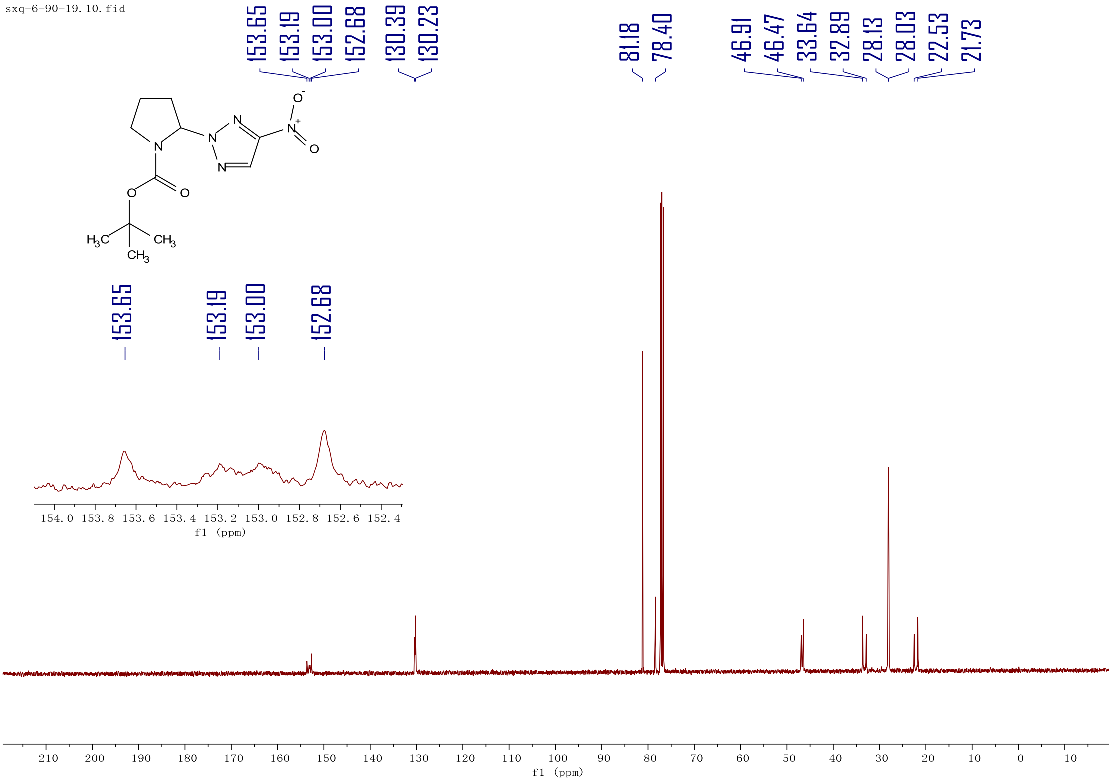
3t (rotameric mixture)



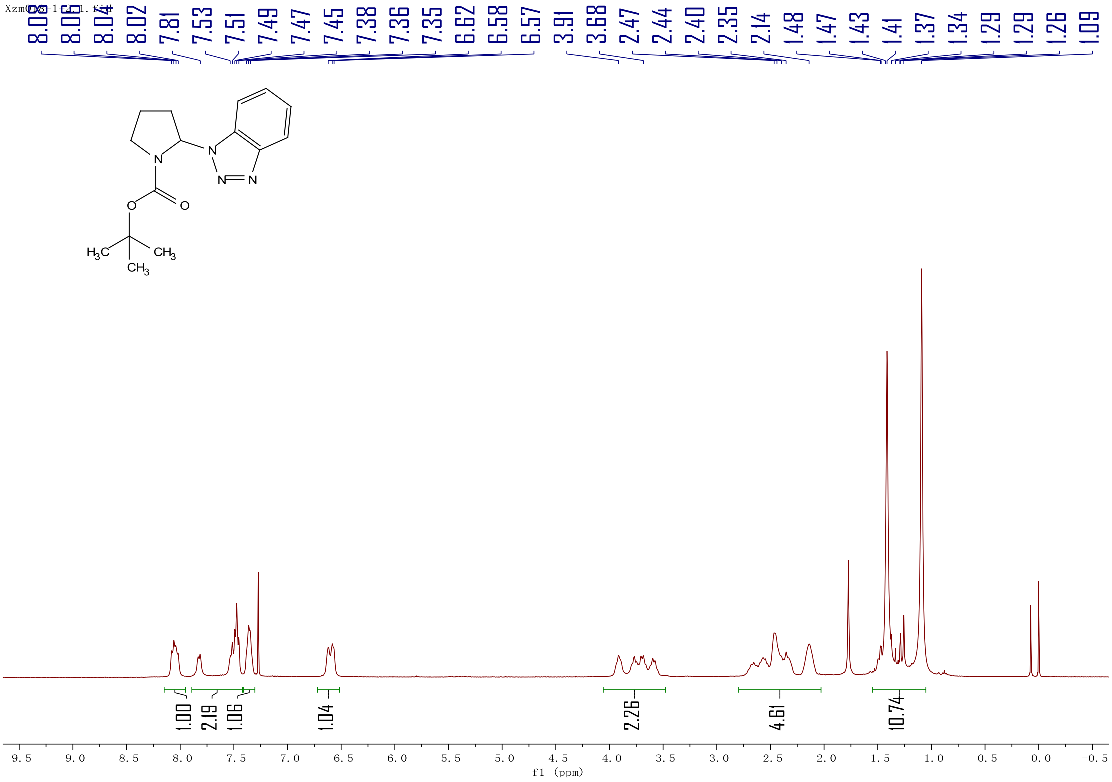


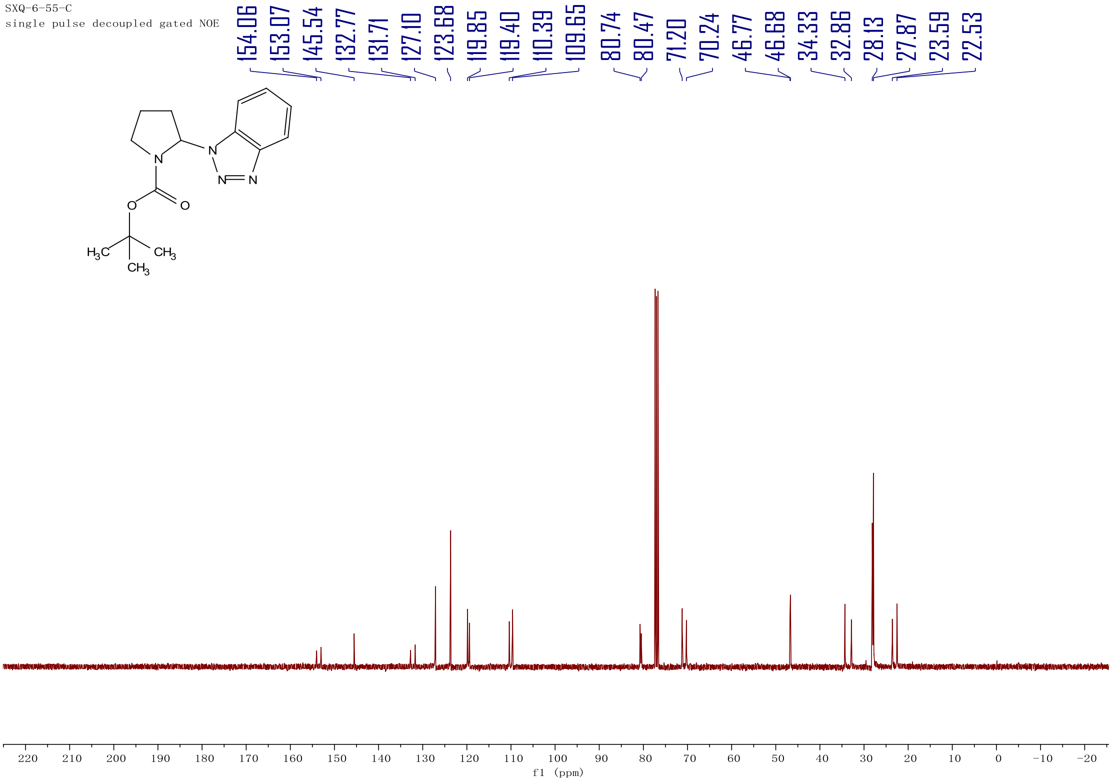
3u (rotameric mixture)



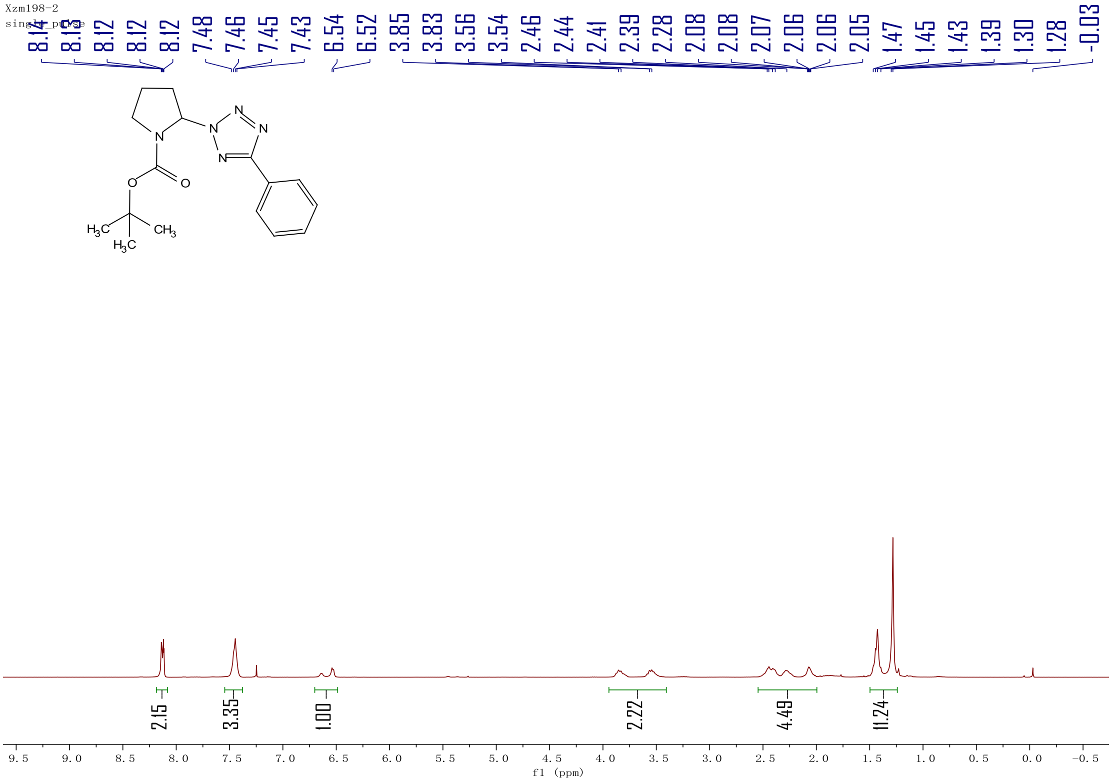


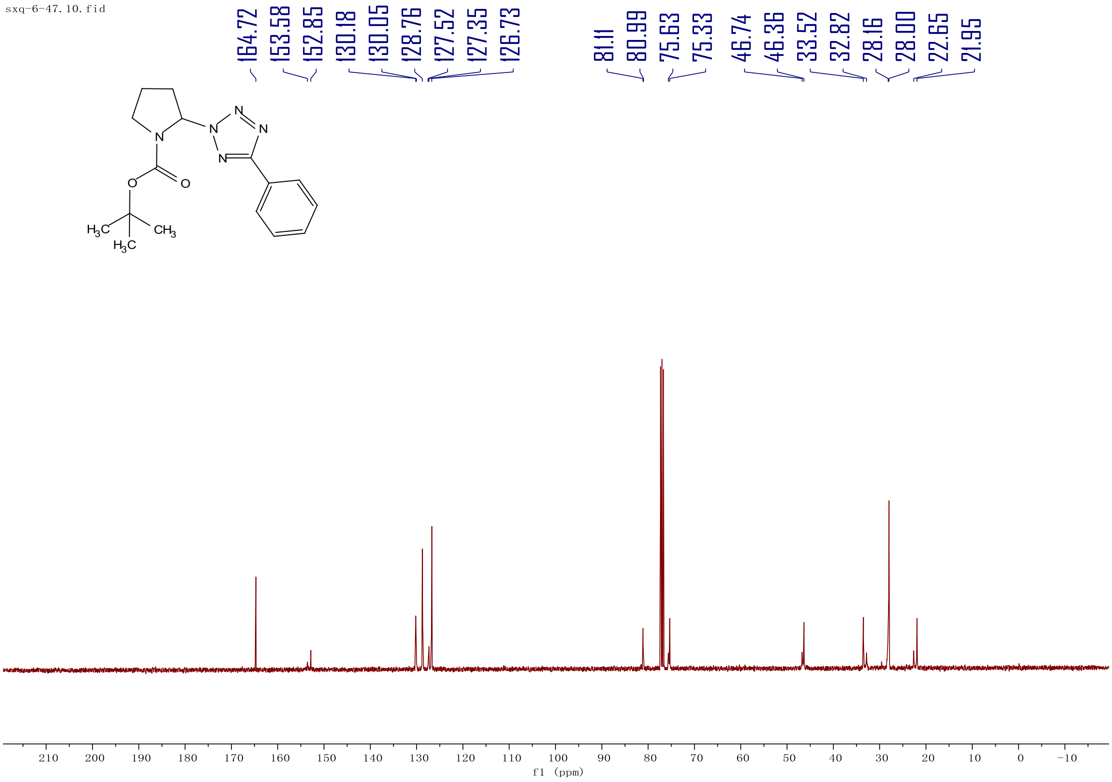
3v (rotameric mixture)



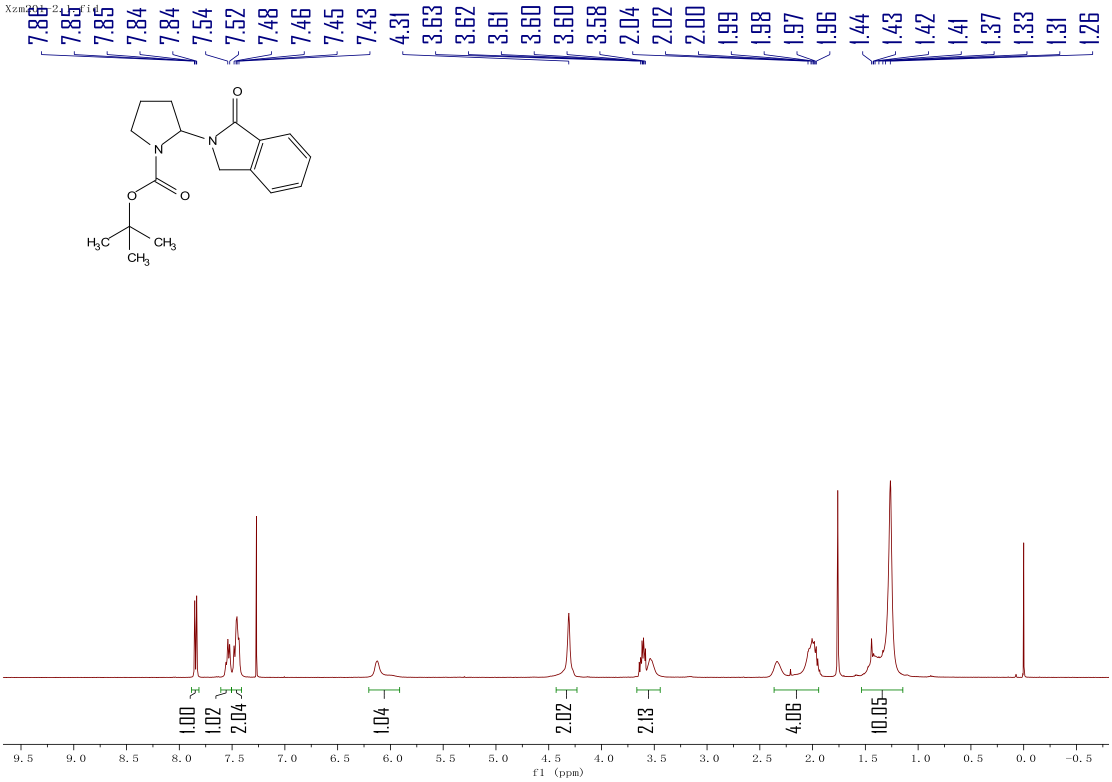


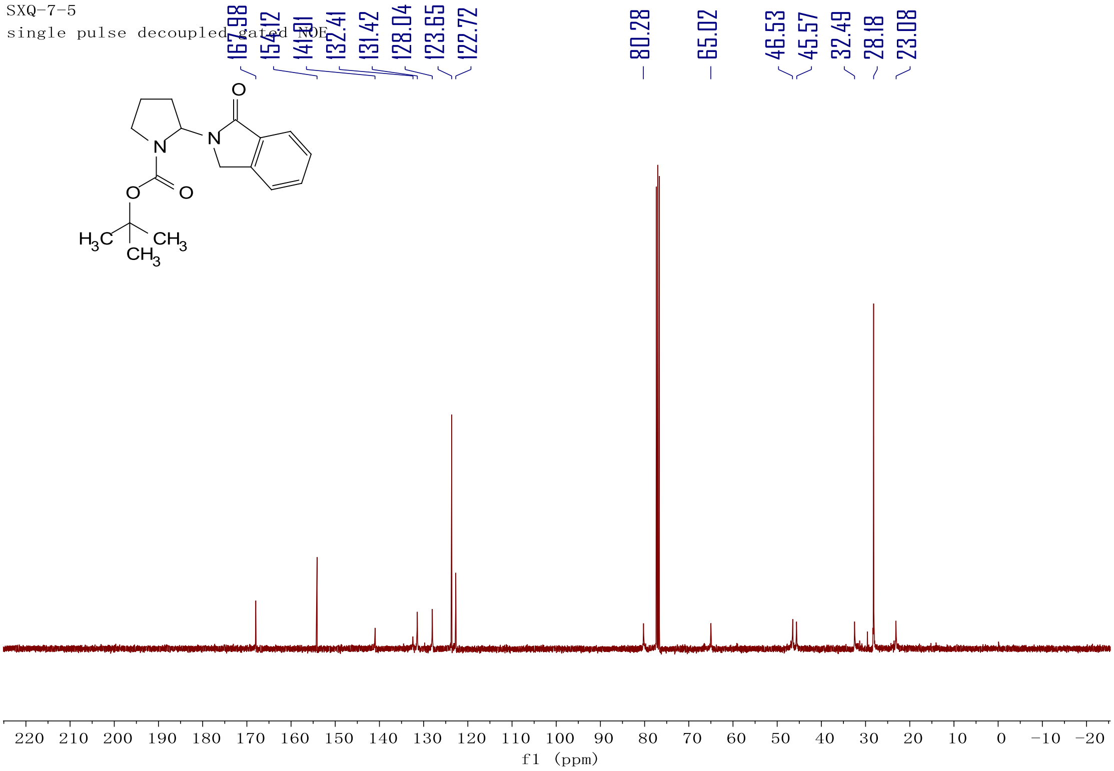
3w (rotameric mixture)



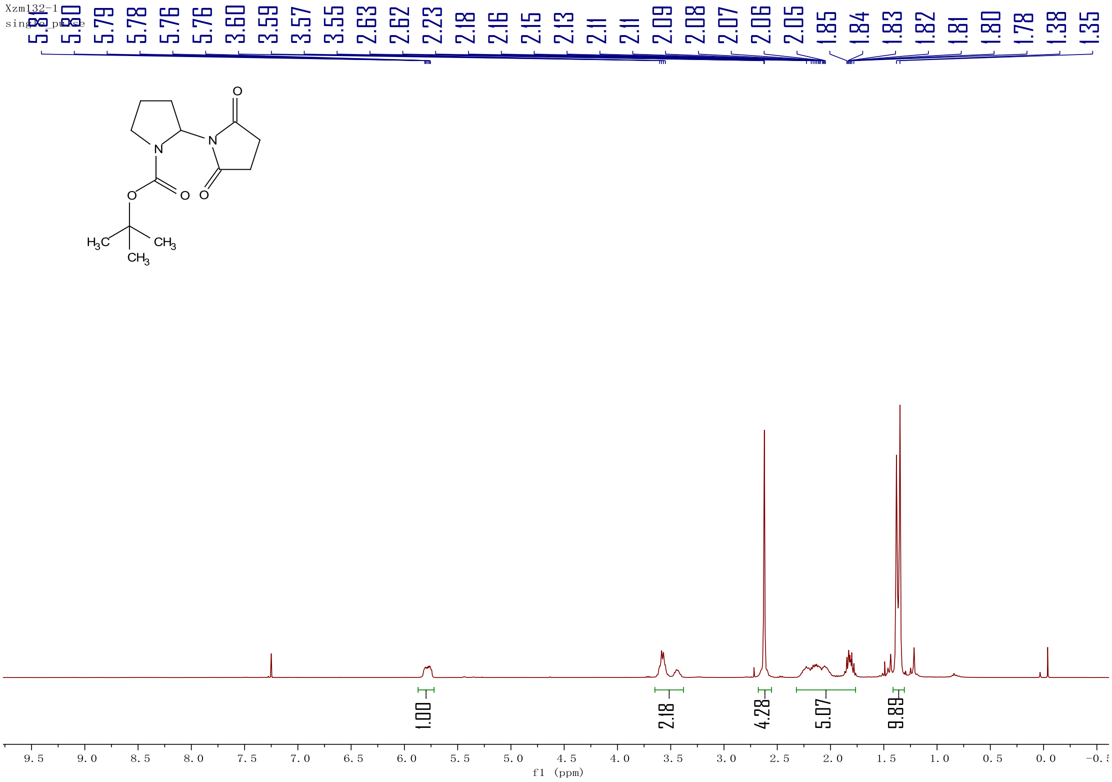


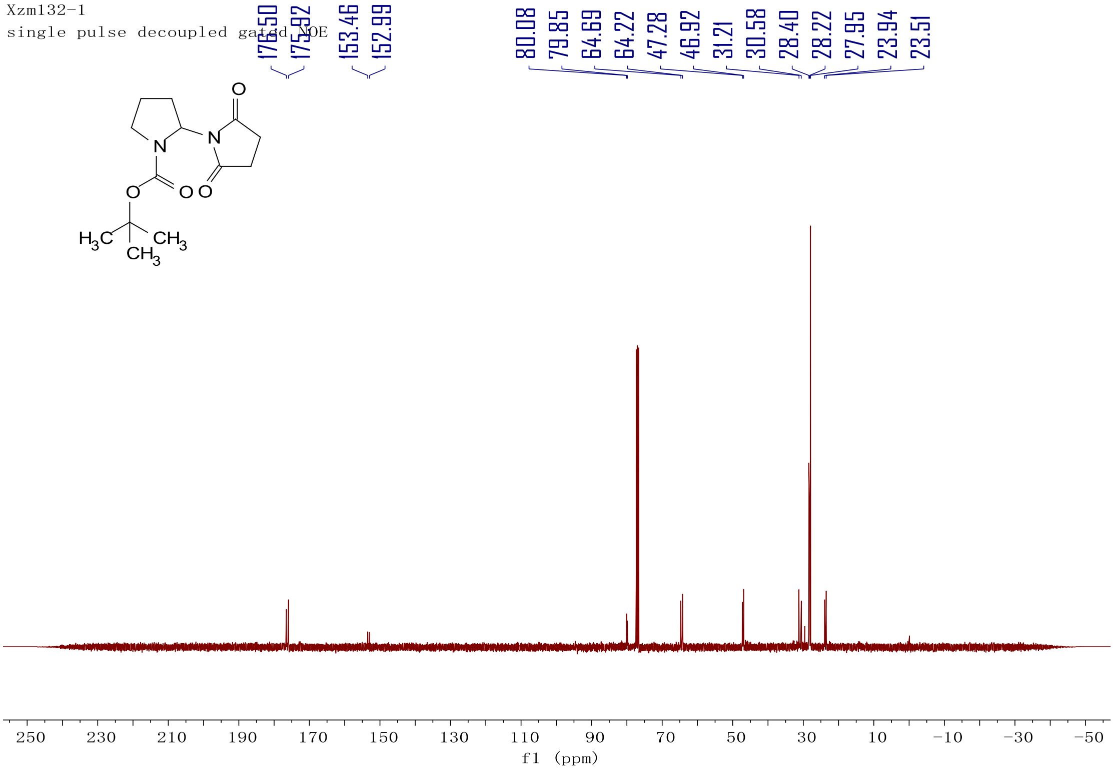
3x


Xzm201-2.1.fid
7.86
7.85
7.85
7.84
7.84
7.54
7.52
7.48
7.46
7.45
7.43
4.31
3.63
3.62
3.61
3.60
3.60
3.58
2.04
2.02
2.00
1.99
1.98
1.97
1.96
1.44
1.43
1.42
1.41
1.37
1.33
1.31
1.26
O
N
N
O
O
H3C
CH3
CH3
1.00
1.02
2.04
1.04
2.02
2.13
4.06
10.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

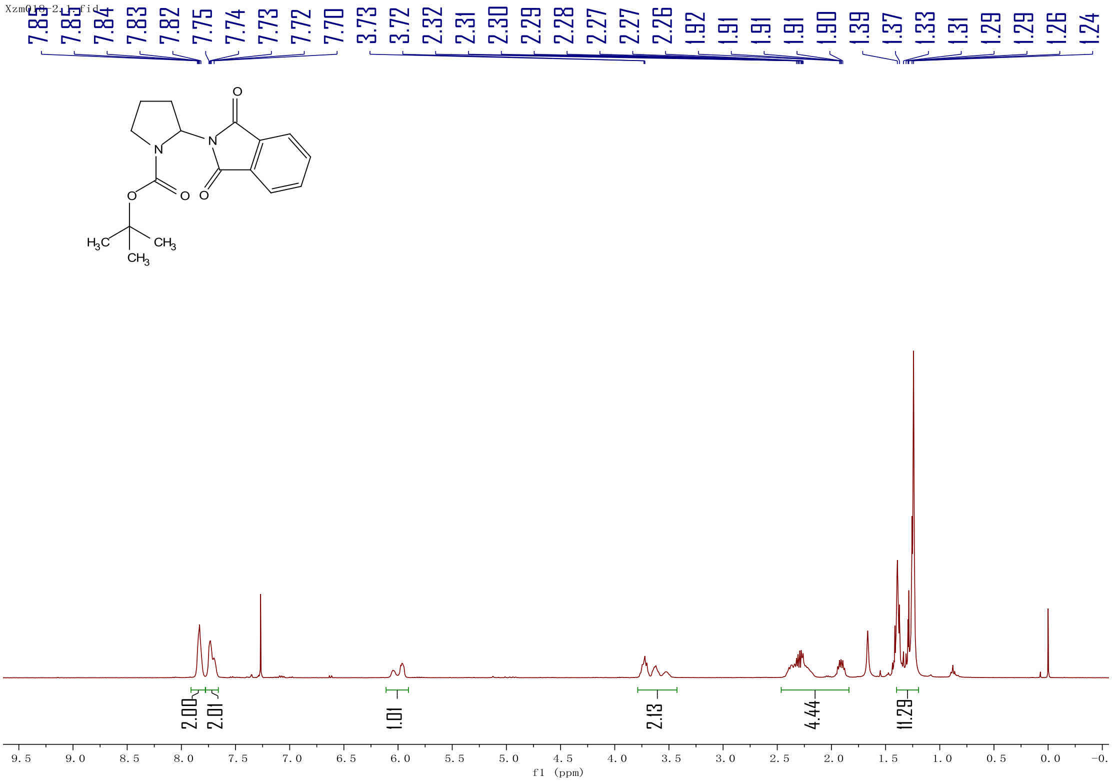

SXQ-7-5
single pulse decoupled gated NOE
167.98
154.12
141.01
132.41
131.42
128.04
123.65
122.72
80.28
65.02
46.53
45.57
32.49
28.18
23.08
O
N
N
O
O
H3C
CH3
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

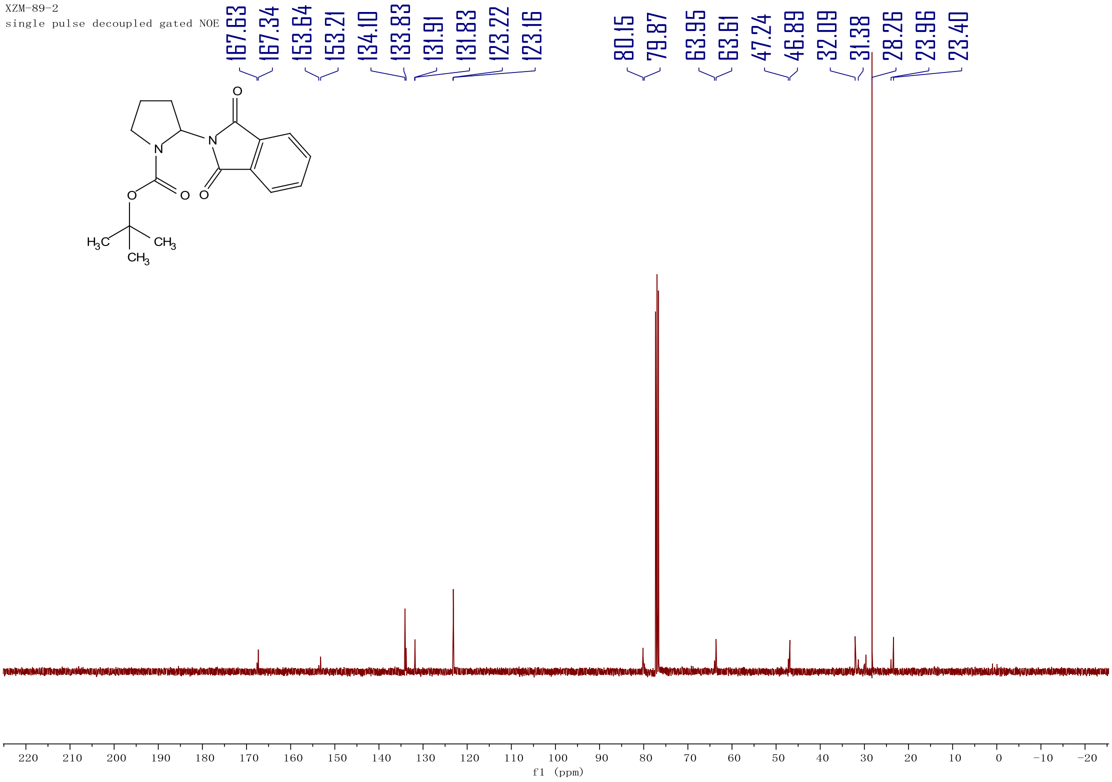
3y (rotameric mixture)



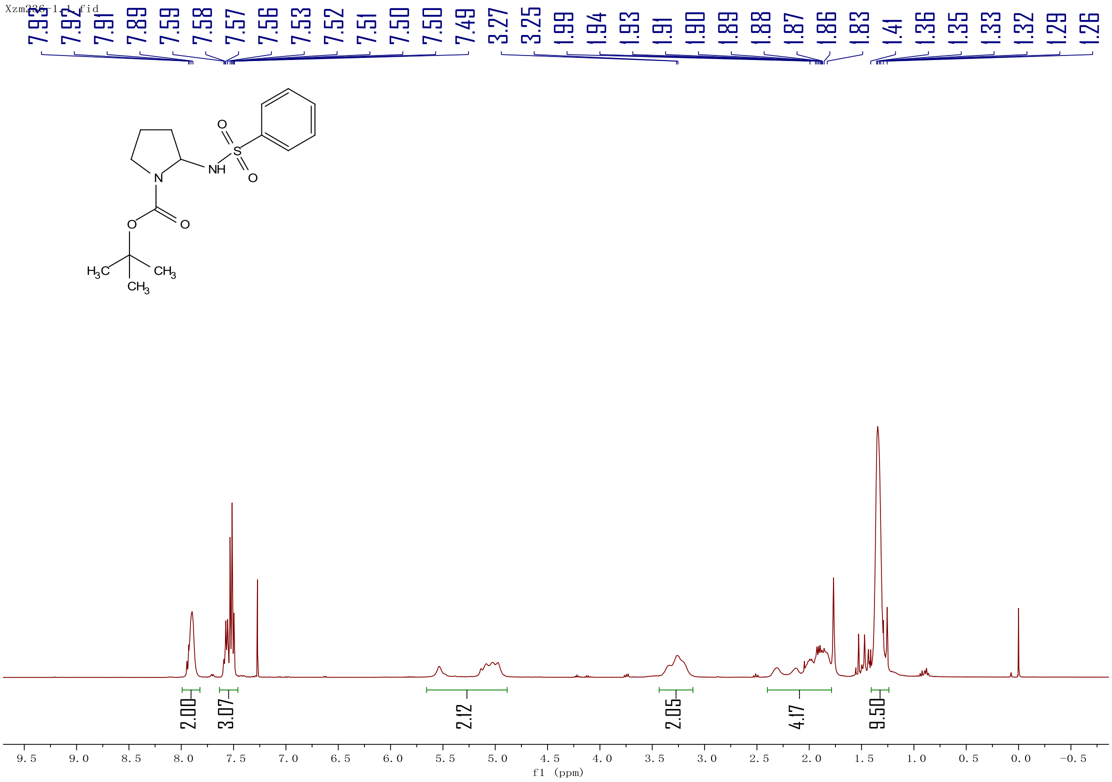


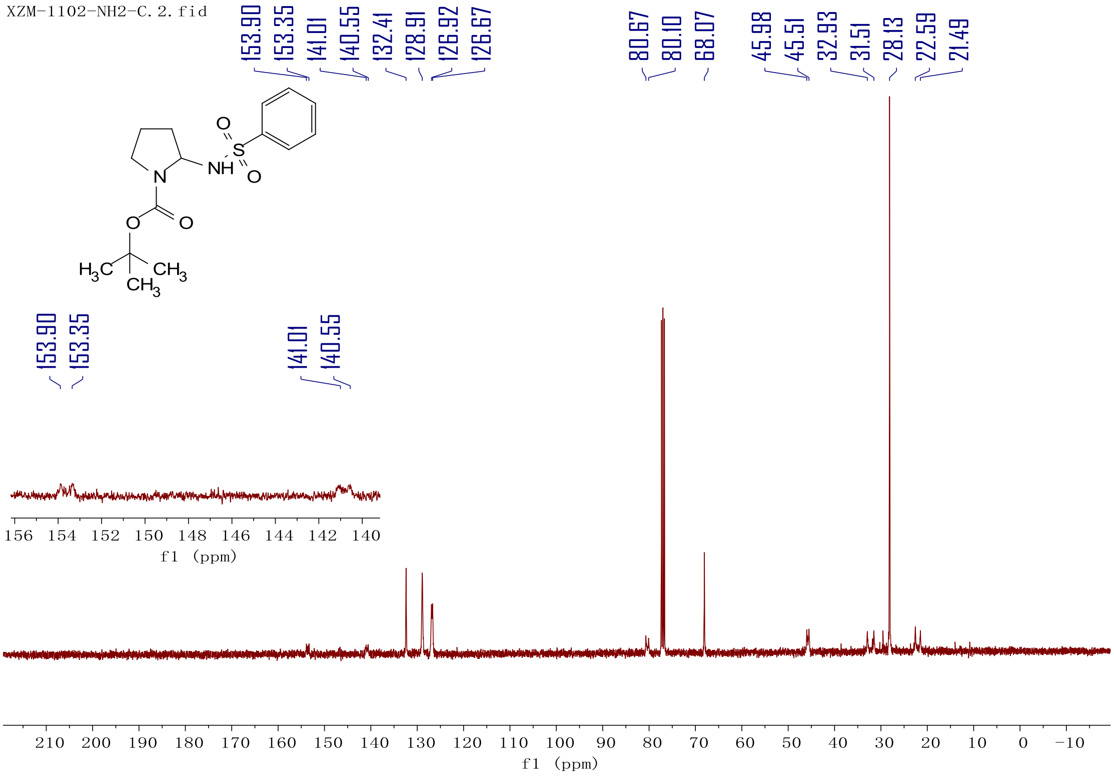
3z (rotameric mixture)



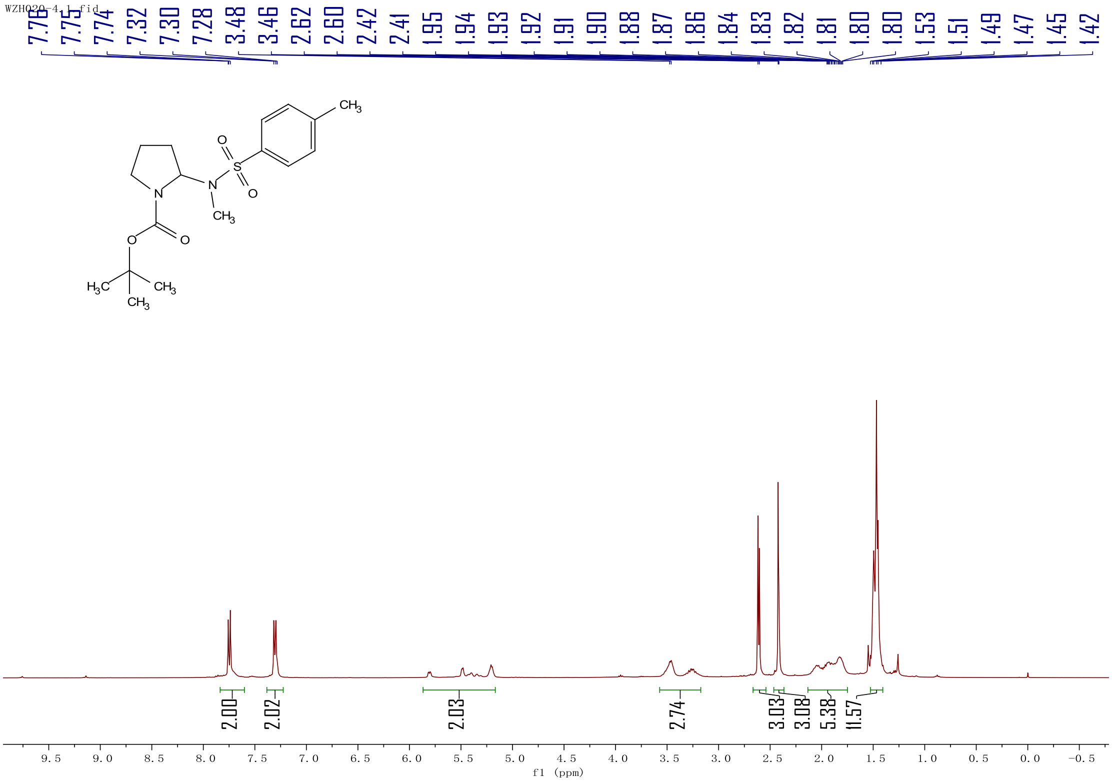


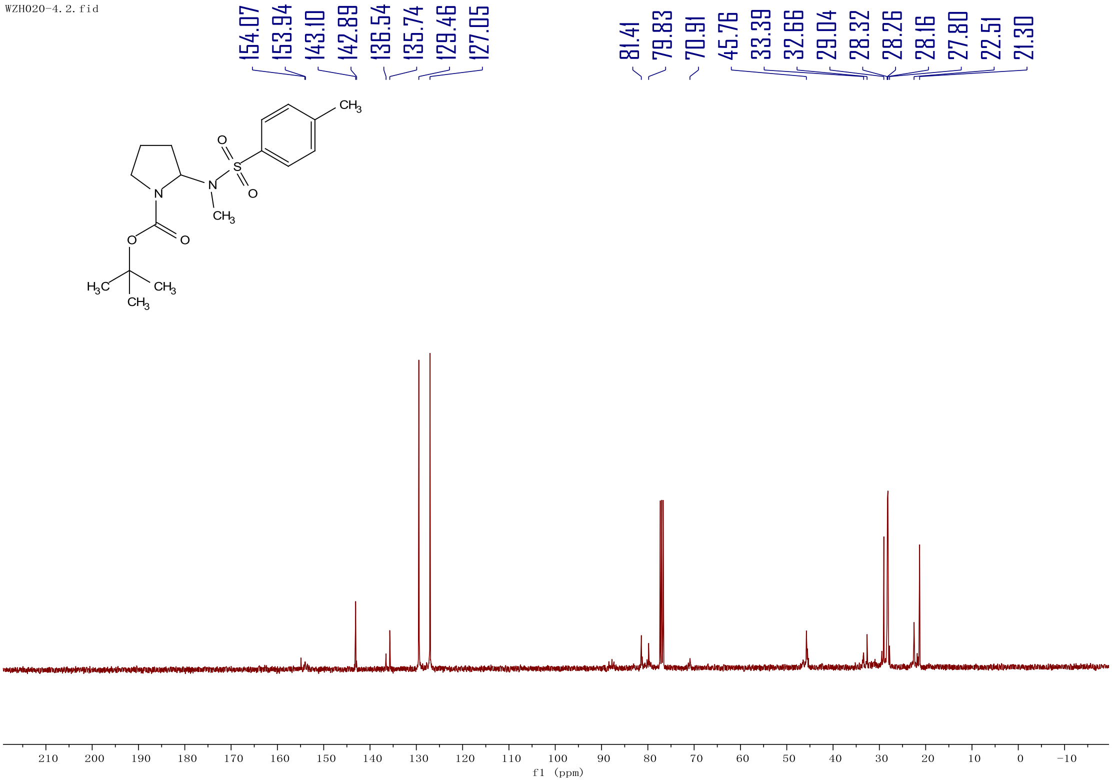
3aa (rotameric mixture)



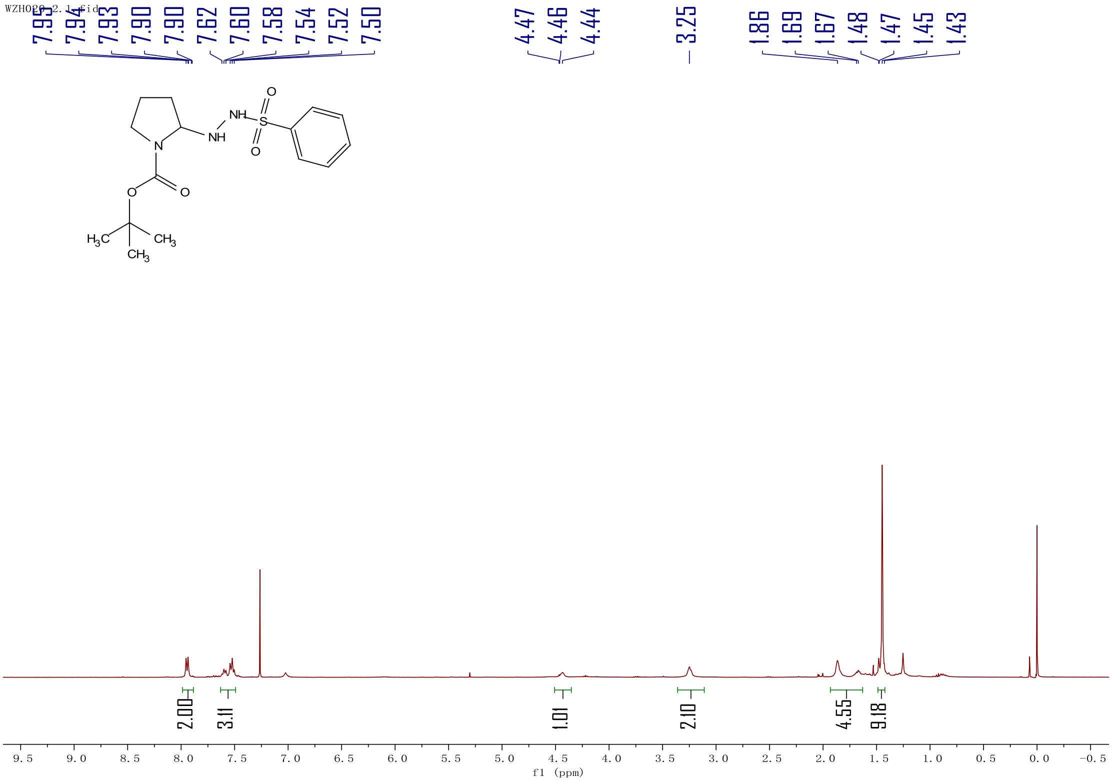


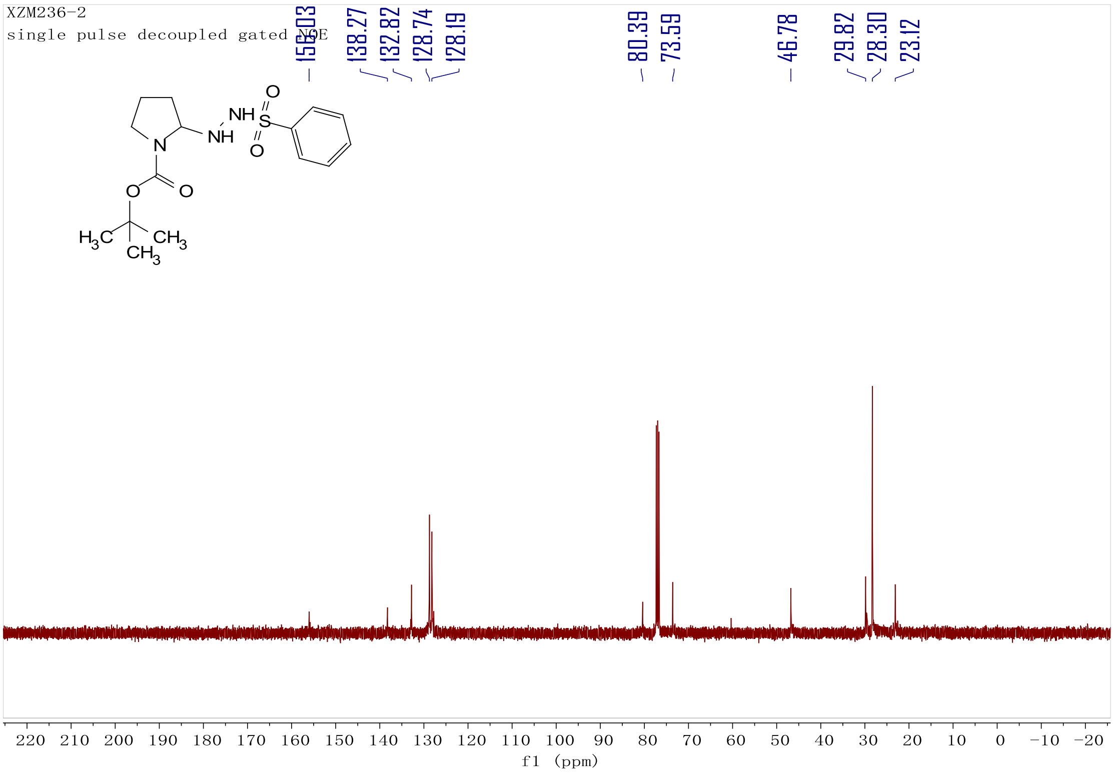
3ab (rotameric mixture)



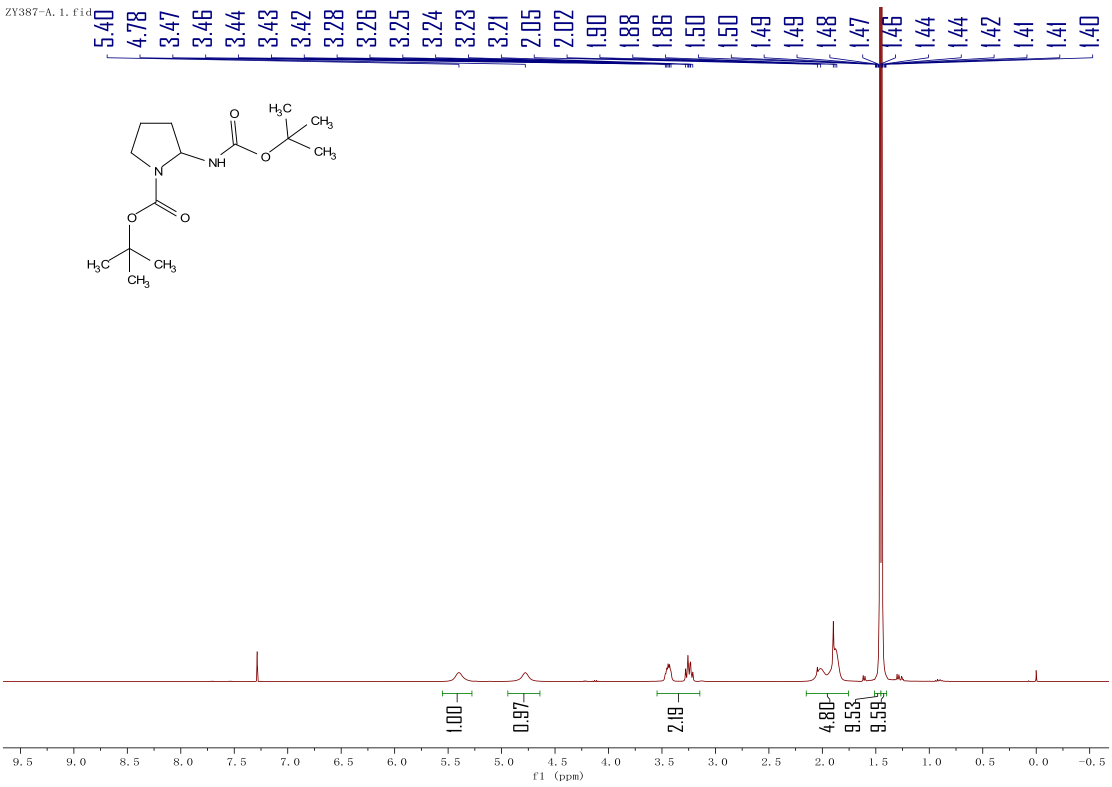


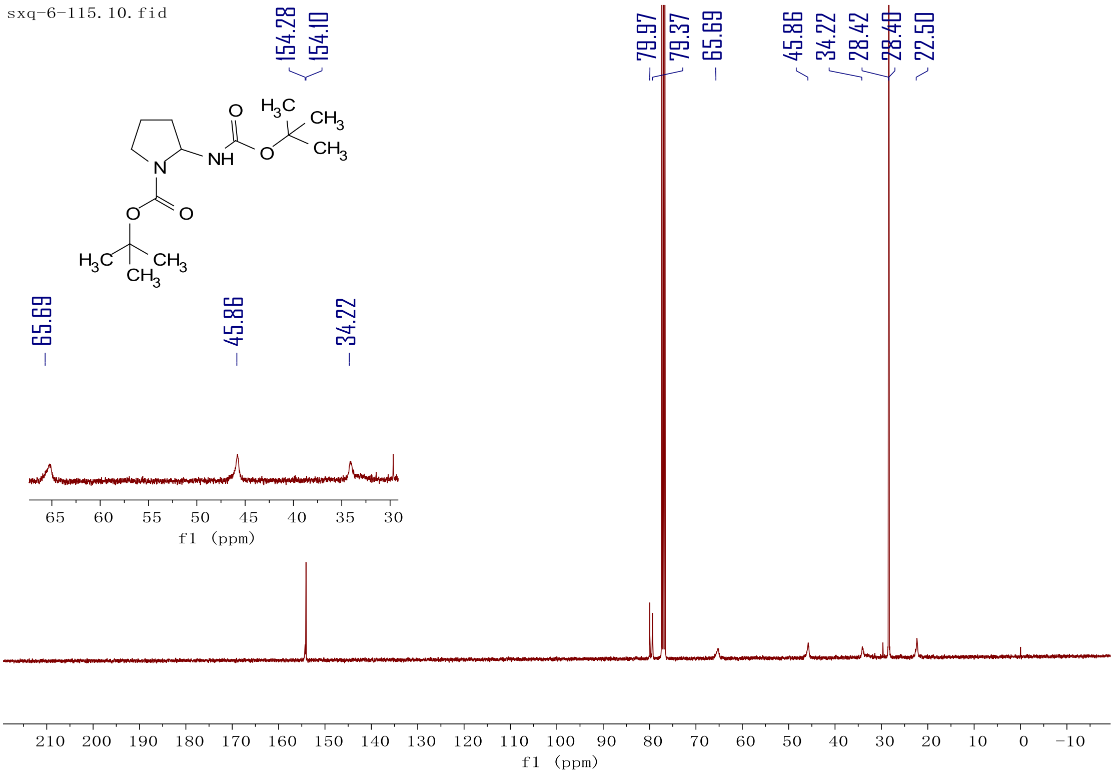
3ac (rotameric mixture)



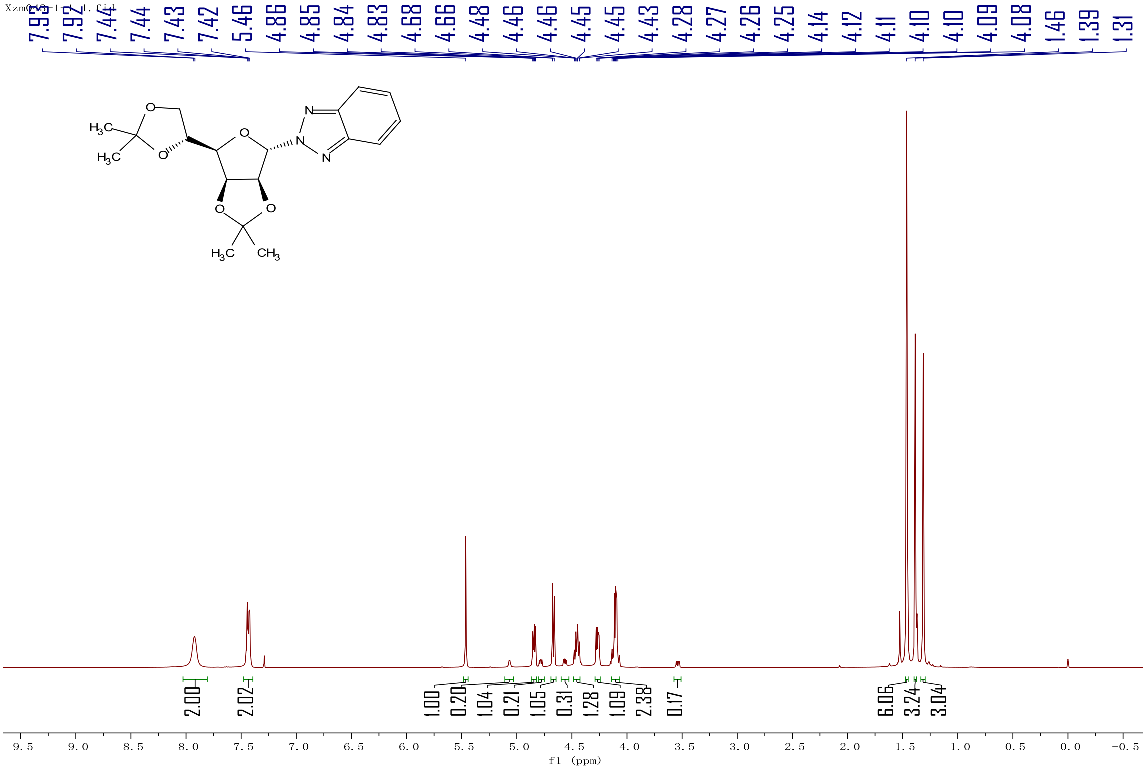


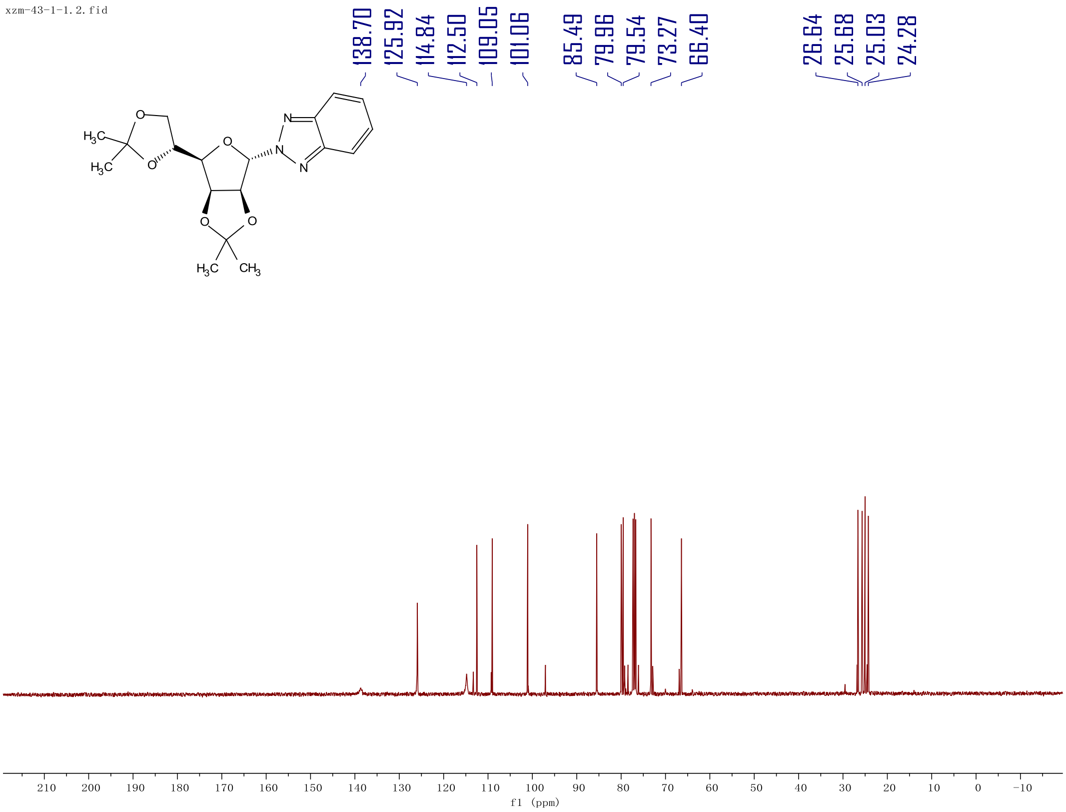
3ad (rotameric mixture)



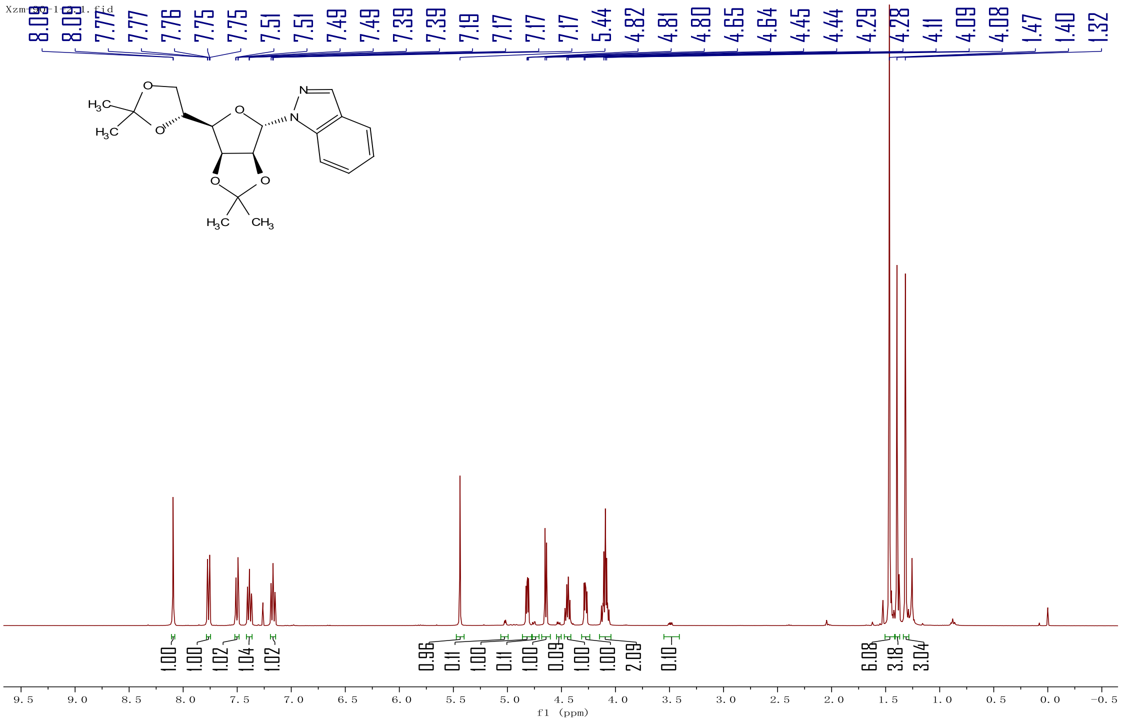


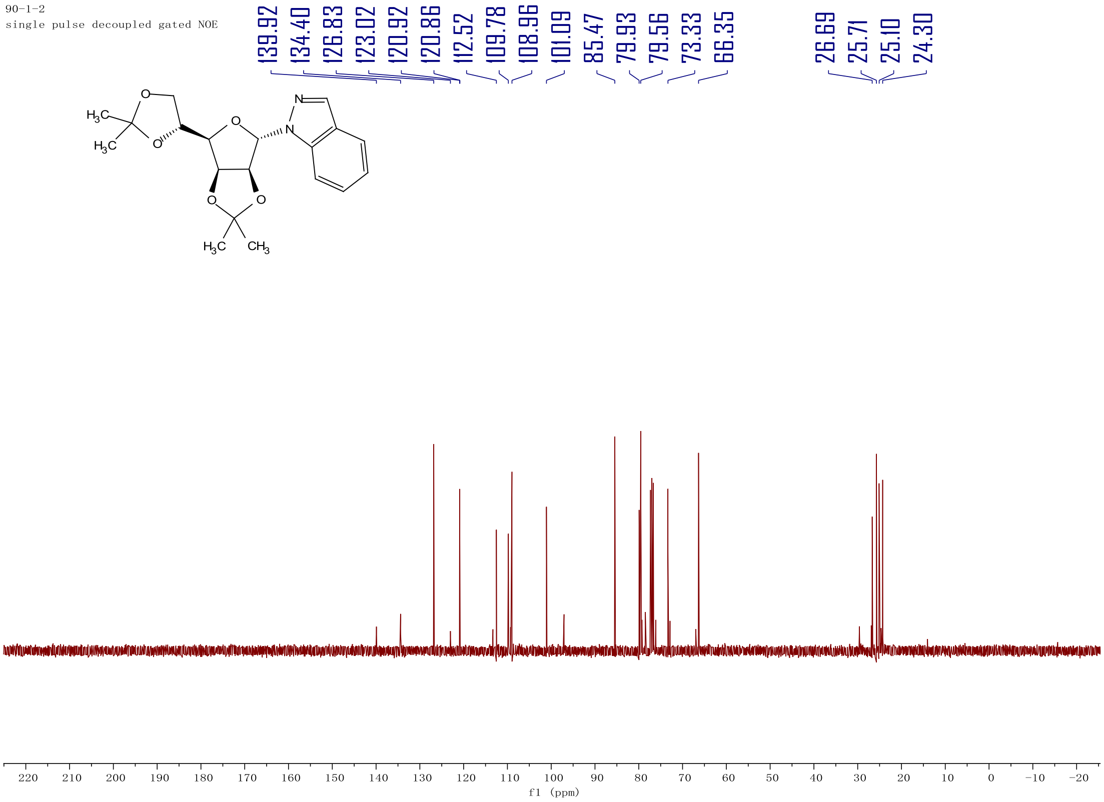
3ae (anomeric mixtures α:β = 5:1)





3af (anomeric mixtures α:β = 10:1)





3ag (anomeric mixtures α:β = 5:1)